

EAST Search History

1 of 2
10/785,229

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L9	1964	502/209 OR 502/305 OR 502/306 OR 502/321 OR 560/231 OR 560/241	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	OFF	2006/03/22 06:22
L10	1071	L9 AND (CARBOXYLIC OR CARBOXYLIC OR CARBOXYLATE OR ACETIC OR PROPIONIC)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L11	974	L10 AND (CATALYST OR HETEROPOLYACID OR TUNGSTATE OR MOLYBDATE OR VANADATE OR SILICOTUNGSTATE OR PHOSPHOVANADATE OR PHOSPHOMOLYBDATE OR PHOSPHOTUNGSTATE OR SILICOPHOSPHOTUNGSTATE OR SILICOPHOSPHOVANADATE OR SILICOPHOSPHOMOLYBDATE OR SILICOPHOSPHOVANDIC OR SILICOPHOSPHOMOLYBDIC OR SILICOPHOSPHOTUNGSTIC OR TUNGSTIC OR MOLYBDIC OR VANDADIC OR SILICOTUNGSTIC OR PHOSPHOVANDADIC OR PHOSPHOMOLYBDIC)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L12	330	L11 AND (OLEFIN OR OLEFINE)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L13	298	L12 AND (GAS OR VAPOR OR VAPOUR)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L14	226	L13 AND (SUPPORT OR SUPPORTED OR SUPPORTING)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L15	221	L14 AND (REACTOR OR COLUMN OR COLUMNAR OR VESSEL OR APPARATUS OR APPARATTUS OR APARATTUS)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22

21

10/785, 229

EAST Search History

L16	143	L15 AND CONTACTING	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L17	6	I16 and (lower adj aliphatic adj carboxylic)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:23
L18	54	I16 and ester	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 08:51
L19	0	I18 not I18	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:23
L20	48	I18 not I17	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:23
L21	10	<i>CLASSE + SUBCLASSED (ON PREV. PAGE) EVERY</i> I9 and (lower adj (olefin or olefine)) and ((carboxylic adj acid adj ester) or (carboxylic adj ester)) and ((gas adj phase) or ((vapor or vapour) adj phase))	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 08:53

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IPC reform
NEWS 4 DEC 23 New IPC8 SEARCH, DISPLAY, and SELECT fields in USPATFULL/
USPAT2
NEWS 5 JAN 13 IPC 8 searching in IFIPAT, IFIUDB, and IFICDB
NEWS 6 JAN 13 New IPC 8 SEARCH, DISPLAY, and SELECT enhancements added to
INPADOC
NEWS 7 JAN 17 Pre-1998 INPI data added to MARPAT
NEWS 8 JAN 17 IPC 8 in the WPI family of databases including WPIFV
NEWS 9 JAN 30 Saved answer limit increased
NEWS 10 JAN 31 Monthly current-awareness alert (SDI) frequency
added to TULSA
NEWS 11 FEB 21 STN AnaVist, Version 1.1, lets you share your STN AnaVist
visualization results
NEWS 12 FEB 22 Status of current WO (PCT) information on STN
NEWS 13 FEB 22 The IPC thesaurus added to additional patent databases on STN
NEWS 14 FEB 22 Updates in EPFULL: IPC 8 enhancements added
NEWS 15 FEB 27 New STN AnaVist pricing effective March 1, 2006
NEWS 16 FEB 28 MEDLINE/LMEDLINE reload improves functionality
NEWS 17 FEB 28 TOXCENTER reloaded with enhancements
NEWS 18 FEB 28 REGISTRY/2REGISTRY enhanced with more experimental spectral
property data
NEWS 19 MAR 01 INSPEC reloaded and enhanced
NEWS 20 MAR 03 Updates in PATDPA: addition of IPC 8 data without attributes
NEWS 21 MAR 08 X.25 communication option no longer available after June 2006
NEWS EXPRESS FEBRUARY 15 CURRENT VERSION FOR WINDOWS IS V8.01a,
CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.00c(JP),
AND CURRENT DISCOVER FILE IS DATED 19 DECEMBER 2005.
V8.0 AND V8.01 USERS CAN OBTAIN THE UPGRADE TO V8.01a AT
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TOTAL SESSION 0.84

FULL ESTIMATED COST

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FILE LAST UPDATED: 21 Mar 2006 (20060321/ED)

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=> S ESTER AND HETEROPOLYACID AND (GAS OR VAPOR OR VAPOUR) AND (OLEFINE OR OLEFIN)
AND (CARBOXYLIC OR ACETIC OR PROPIONIC OR ACRYLIC OR METHACRYLIC)

575051 ESTER

427684 ESTERS

802811 ESTER

(ESTER OR ESTERS)

1348 HETEROPOLYACID

936 HETEROPOLYACIDS

1913 HETEROPOLYACID

(HETEROPOLYACID OR HETEROPOLYACIDS)

1485523 GAS

503872 GASES

1665041 GAS

(GAS OR GASES)

504059 VAPOR

70554 VAPORS

545733 VAPOR

(VAPOR OR VAPORS)

2270 VAPOUR

187 VAPOURS

2448 VAPOUR

(VAPOUR OR VAPOURS)

108 OLEFINE

250 OLEFINS

355 OLEFINE

(OLEFINE OR OLEFINS)

97518 OLEFIN

100671 OLEFINS

152638 OLEFIN

(OLEFIN OR OLEFINS)

236013 CARBOXYLIC

47 CARBOXYLICS

236032 CARBOXYLIC

(CARBOXYLIC OR CARBOXYLICS)

221349 ACETIC

STN SEARCH TRANSCRIPT

10/785,229

OTHER SOURCE(S): WO 2002-JP11683 W 20021108
WO 2002-JP11686 W 20021108
CASREACT 140:43772
AB Lower aliphatic carboxylic acid esters (e.g., Et acetate) are prepared by esterifying a lower aliphatic carboxylic acid and a lower olefin (e.g., ethylene) into a lower aliphatic carboxylic acid ester using an acid catalyst in a vapor phase, when the system is controlled to contain substantially no acetylene compounds, the deterioration of the catalyst can be remarkably prevented from proceeding and in turn a stable operation can be continuously performed for a long time. Examples of the acid catalyst which can be used in the present invention include compounds widely known in general as an acid catalyst, such as a heteropolyacid and a salt; process flow diagrams are presented.
REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L1 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2000:68415 CAPLUS
DOCUMENT NUMBER: 132:94968
TITLE: Process and catalysts for the manufacture of esters by the addition reaction of lower alkenes with aliphatic monocarboxylic acids with removal of nitrogenous bases from the reactants
INVENTOR(S): Coker, Eric Nicholas; Froom, Simon Frederick Thomas; Smith, Warren John
PATENT ASSIGNEE(S): BP Chemicals Limited, UK
SOURCE: PCT Int. Appl., 16 pp.
CODEN: PFXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
WO 200003966 A1 20000127 WO 1999-GB2099 19990701
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IN, IS, JP, KE, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NA, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BE, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
CA 2336946 AA 20000127 CA 1999-2336946 19990701
AU 9946323 A1 20000207 AU 1999-46323 19990701
BR 9912038 A 20010403 BR 1999-12038 19990701
EP 1097120 A1 20010509 EP 1999-929533 19990701
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO
JP 2002520380 T2 20020709 JP 2000-560076 19990701
TW 502015 B 20020911 TW 1999-88111867 19990713
US 2001047107 A1 20011129 US 2001-752835 20010103
ZA 2001000331 A 20020111 GB 1998-15117 A 19980714
WO 1999-GB2099 19990701
PRIORITY APPLN. INFO.: WO 1999-GB2099 19990701
AB Lower aliphatic esters are prepared in high yield and selectivity by the addition reaction of a lower olefin (e.g., ethylene) with a saturated lower aliphatic monocarboxylic acid in the vapor phase in the presence of a heteropolyacid catalyst; the reactants are rendered substantially free of basic, nitrogen compounds, by contact with an acidic, heterogeneous solid prior to being brought into contact with the heteropoly acid catalyst.

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
L1 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1997:192121 CAPLUS
DOCUMENT NUMBER: 126:185811
TITLE: Process and heteropolyacid catalysts for the carboxylic acids

INVENTOR(S): Atkins, Martin Philip; Sharma, Bhushan
PATENT ASSIGNEE(S): BP Chemicals Limited, UK
SOURCE: Eur. Pat. Appl., 23 pp.
CODEN: EPXDBW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
EP 757027 A1 19970205 EP 1996-305369 19960722
EP 757027 B1 20000405
R: BE, DE, FR, GB, NL, SE
EP 959064 A1 19991124 EP 1999-113642 19960722
EP 959064 B1 20011212
R: BE, DE, FR, GB, NL, SE
US 5861530 A 19990119
CA 2182558 AA 19970203
JP 09118647 A2 19970506
CN 1150585 A 19970528
CN 1085197 B 20020522
JP 2005298527 A2 20051027
PRIORITY APPLN. INFO.:

AB Lower aliphatic esters (e.g., EtO2CCH3) are prepared by reacting a lower olefin (e.g., H2C=CH2) with a saturated lower aliphatic monocarboxylic acid (e.g., AcOH) in the vapor phase in the presence of a heteropolyacid catalyst, characterized in that an amount of water in the range from 1-10 mol% (based on the total of olefin, aliphatic monocarboxylic acid and water) is added to the reaction mixture during the reaction. The presence of water enhances the ester yield. The reaction mixture may optionally contain a dielectric (e.g., EtOEt) to minimize byproduct formation.

L1 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1994:579103 CAPLUS
DOCUMENT NUMBER: 121:179103
TITLE: Preparation of carboxylate esters
INVENTOR(S): Nagamura, Hiroo; Yagi, Hirobumi; Morita, Takehiko; Sugimura, Toshiro
PATENT ASSIGNEE(S): Nippon Catalytic Chem Ind, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 12 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
JP 06072951 A2 19940315 JP 1993-76705 19930402
PRIORITY APPLN. INFO.: JP 1992-180824 A1 19920708

AB The title compds. are prepared by gas-phase treating carboxylic acids with olefins in the presence of O and acid salts of heteropoly acids. A reactor containing HO.5Cs2.5PW12O40 (preparation given) was fed with a 1:13:0.02 mixed gases of acrylic acid, CH2:CH2, and O at 150° and atmospheric for 3 h to give 49.7% Et acrylate.

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(FILE 'HOME' ENTERED AT 09:10:45 ON 22 MAR 2006)

L1 FILE 'CAPLUS' ENTERED AT 09:12:50 ON 22 MAR 2006
5 S ESTER AND HETEROPOLYACID AND (GAS OR VAPOR OR VAPOUR) AND (OL OR VAPOUR) AND (CARRIER OR SUPPORT OR SUPPORTING OR SUPPORTED) AND (GAS OR VAPOR OR VAPOUR) AND (OLEFINE OR OLEFIN) AND (CARBOXYLIC OR ACETIC OR PROPIONIC OR ACRYLIC OR METHACRYLIC)

=> S ESTER AND (CARRIER OR SUPPORT OR SUPPORTING OR SUPPORTED) AND (GAS OR VAPOR OR VAPOUR) AND (OLEFINE OR OLEFIN) AND (CARBOXYLIC OR ACETIC OR PROPIONIC OR ACRYLIC OR METHACRYLIC)

575051 ESTER
427684 ESTERS
802811 ESTER
264065 CARRIER
147441 CARRIERS
345441 CARRIER
441539 SUPPORT
123551 SUPPORTS
524635 SUPPORT
89557 SUPPORTING
5 SUPPORTINGS
89561 SUPPORTING (SUPPORTING OR SUPPORTINGS)
197847 SUPPORTED
1485523 GAS
503872 GASES
1665041 GAS (GAS OR GASES)
504059 VAPOR
70554 VAPORS
545733 VAPOR (VAPOR OR VAPORS)
2270 VAPOUR
187 VAPOURS
2448 VAPOUR (VAPOUR OR VAPOURS)
108 OLEFINE
250 OLEFINES
355 OLEFINE (OLEFINE OR OLEFINS)
97518 OLEFIN
100671 OLEFINS
152638 OLEFIN (OLEFIN OR OLEFINS)
236013 CARBOXYLIC
47 CARBOXYLICS

236032 CARBOXYLIC (CARBOXYLIC OR CARBOXYLICS)
221349 ACETIC
22 ACETICS
221358 ACETIC (ACETIC OR ACETICS)
54670 PROPIONIC
8 PROPIONICS
54674 PROPIONIC (PROPIONIC OR PROPIONICS)
258085 ACRYLIC
1322 ACRYLICS
258457 ACRYLIC (ACRYLIC OR ACRYLICS)
73789 METHACRYLIC
6 METHACRYLICS
73793 METHACRYLIC (METHACRYLIC OR METHACRYLICS)
L2 53 ESTER AND (CARRIER OR SUPPORT OR SUPPORTING OR SUPPORTED) AND (GAS OR VAPOR OR VAPOUR) AND (OLEFINE OR OLEFIN) AND (CARBOXYLIC OR ACETIC OR PROPIONIC OR ACRYLIC OR METHACRYLIC)

=> S L2 AND (SILICOTUNGSTIC OR PHOSPHOTUNGSTIC OR PHOSPHOMOLYBDIC OR SILICOMOLYBDIC OR SILICOVANADOTUNGSTIC OR PHOSPHOVANADOTUNGSTIC OR PHOSPHOVANADOMOLYBDIC)

1331 SILICOTUNGSTIC
4744 PHOSPHOTUNGSTIC
2773 PHOSPHOMOLYBDIC
1169 SILICOMOLYBDIC
1 SILICOVANADOTUNGSTIC
11 PHOSPHOVANADOTUNGSTIC
57 PHOSPHOVANADOMOLYBDIC
L3 1 L2 AND (SILICOTUNGSTIC OR PHOSPHOTUNGSTIC OR PHOSPHOMOLYBDIC OR SILICOMOLYBDIC OR SILICOVANADOTUNGSTIC OR PHOSPHOVANADOMOLYBDIC)

=> D

L3 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2004:1036764 CAPLUS
DN 142:40422
TI Support and catalyst for use in producing lower aliphatic carboxylic acid ester, catalyst manufacture process and method of use
IN Kadowaki, Etsuko; Higashi, Tomoyoshi; Oguchi, Wataru; Uchida, Hiroshi;
PA Narumi, Kousuke
SO Showa Denko K.K., Japan
U.S. Pat. Appl. Publ., 27 pp., Cont.--in-part of U.S. Ser. No. 70,259, abandoned.
CODEN: USXXCO
DT Patent
LA English
FAM.CNT 3

PATENT NO. KIND DATE APPLICATION NO. DATE

PI US 2004242918 A1 20041202 US 2004-785229 20040225
JP 2002079090 A2 20020319 JP 2001-173624 20010608
WO 2002000589 A2 20020103 WO 2001-075532 20010627
WO 2002000589 A3 20020418

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MX, MY, NA, NI, NL, NO, NZ, PA, PE, PG, PH, PL, PT, RO, RU, SD, SE, SI, SK, SL, SM, SN, ST, SV, SW, SZ, TC, TD, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AA, AB, AC, AD, AE, AF, AG, AH, AI, AJ, AK, AL, AM, AN, AO, AP, AQ, AR, AS, AT, AU, AV, AW, AX, AY, AZ, BA, BB, BC, BD, BE, BF, BG, BH, BI, BJ, BK, BL, BM, BN, BO, BP, BQ, BR, BS, BT, BU, BV, BW, BY, BZ, CA, CB, CC, CD, CE, CF, CG, CH, CI, CK, CL, CM, CN, CO, CP, CQ, CR, CS, CU, CV, CW, CX, CY, CZ, DD, DE, DF, DG, DH, DI, DJ, DK, DL, DM, DN, DO, DP, DQ, DR, DS, DT, DU, DV, DW, DX, DY, DZ, EA, EB, EC, ED, EE, EF, EG, EH, EI, EJ, EK, EL, EM, EN, EO, EP, EQ, ER, ES, ET, EU, EV, EW, EX, EY, EZ, FA, FB, FC, FD, FE, FF, FG, FH, FI, FJ, FK, FL, FM, FN, FO, FP, FQ, FR, FS, FT, FU, FV, FW, FX, FY, FZ, GA, GB, GC, GD, GE, GF, GH, GI, GJ, GK, GL, GM, GN, GO, GP, GQ, GR, GS, GT, GU, GV, GW, GX, GY, HZ, HA, HB, HC, HD, HE, HF, HG, HH, HI, HJ, HK, HL, HM, HN, HO, HP, HQ, HR, HS, HT, HU, HV, HW, HX, HY, HZ, IA, IB, IC, ID, IE, IF, IG, IH, II, IJ, IK, IL, IM, IN, IO, IP, IQ, IR, IS, IT, IU, IV, IW, IX, IY, IZ, JA, JB, JC, JD, JE, JF, JG, JH, JI, JJ, JK, JL, JM, JN, JO, JP, JQ, JR, JS, JT, JU, JV, JW, JX, JY, JZ, KA, KB, KC, KD, KE, KF, KG, KH, KI, KJ, KK, KL, KM, KN, KO, KP, KQ, KR, KS, KT, KU, KV, KW, KX, KY, KZ, LA, LB, LC, LD, LE, LF, LG, LH, LI, LJ, LK, LM, LN, LO, LP, LQ, LR, LS, LT, LU, LV, LW, LX, LY, LZ, MA, MB, MC, MD, ME, MF, MG, MH, MI, MJ, MK, ML, MN, MO, MP, MQ, MR, MS, MT, MU, MV, MW, MX, MY, MZ, NA, NB, NC, ND, NE, NF, NG, NH, NI, NJ, NK, NL, NM, NO, NP, NQ, NR, NS, NT, NU, NV, NW, NX, NY, NZ, OA, OB, OC, OD, OE, OF, OG, OH, OI, OJ, OK, OL, OM, ON, OO, OP, OQ, OR, OS, OT, OU, OV, OW, OX, OY, OZ, PA, PB, PC, PD, PE, PF, PG, PH, PI, PJ, PK, PL, PM, PN, PO, PP, PQ, PR, PS, PT, PU, PV, PW, PX, PY, PZ, QA, QB, QC, QD, QE, QF, QG, QH, QI, QJ, QK, QL, QM, QN, QO, QP, QQ, QR, QS, QT, QU, QV, QW, QX, QY, QZ, RA, RB, RC, RD, RE, RF, RG, RH, RI, RJ, RK, RL, RM, RN, RO, RP, RQ, RR, RS, RT, RU, RV, RW, RX, RY, RZ, SA, SB, SC, SD, SE, SF, SG, SH, SI, SJ, SK, SL, SM, SN, SO, SP, SQ, SR, SS, ST, SU, SV, SW, SX, SY, SZ, TA, TB, TC, TD, TE, TF, TG, TH, TI, TJ, TK, TL, TM, TN, TO, TP, TQ, TR, TS, TT, TU, TV, TW, TX, TY, TZ, UA, UB, UC, UD, UE, UF, UG, UH, UI, UJ, UK, UL, UM, UN, UO, UP, UQ, UR, US, UT, UV, UW, UX, UY, UZ, VA, VB, VC, VD, VE, VF, VG, VH, VI, VJ, VK, VL, VM, VN, VO, VP, VQ, VR, VS, VT, VU, VV, VW, VX, VY, VZ, WA, WB, WC, WD, WE, WF, WG, WH, WI, WJ, WK, WL, WM, WN, WO, WP, WQ, WR, WS, WT, WU, WV, WX, WY, WZ, XA, XB, XC, XD, XE, XF, XG, XH, XI, XJ, XK, XL, XM, XN, XO, XP, XQ, XR, XS, XT, XU, XV, XW, XX, XY, XZ, YA, YB, YC, YD, YE, YF, YG, YH, YI, YJ, YK, YL, YM, YN, YO, YP, YQ, YR, YS, YT, YU, YV, YW, YX, YY, YZ, ZA, ZB, ZC, ZD, ZE, ZF, ZG, ZH, ZI, ZJ, ZK, ZL, ZM, ZN, ZO, ZP, ZQ, ZR, ZS, ZT, ZU, ZV, ZW, ZX, ZY, ZZ.

APPLICANTS

DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
BJ, CF, CG, CI, CM, GA, GW, ML, MR, NE, SN, TD, TG 20010717
US 2003032837 A1 20030213 US 2001-889354 20011207
JP 2002316048 A2 20021029 JP 2001-373675 20020212
WO 2002064541 A1 20020822 WO 2002-JP1156 20020212
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
GM, GR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT,
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RO, RU, SD, SE, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG,
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US 2003118497 A1 20030626 US 2002-70259 20021203
ZA 2002009804 A 20030819 ZA 2002-9804 20030702
PRAI JP 2000-192964 A 20040312 ZA 2003-5143 20030702
US 2000-218803P P 20000718
JP 2001-36038 A 20010213
US 2001-273343P P 20010306
WO 2001-JP5532 W 20010627
US 2001-889354 B2 20010717
JP 2001-373675 A 20011207
WO 2002-JP1156 W 20020212
US 2002-70259 B2 20020304
CASREACT 142:40422

=> S L2 AND HETEROPOLYACID
1348 HETEROPOLYACID
936 HETEROPOLYACID
1913 HETEROPOLYACID
(HETEROPOLYACID OR HETEROPOLYACIDS)
1 L2 AND HETEROPOLYACID

=> D
L4 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2004:1036764 CAPLUS
DN 142:40422
TI support and catalyst for use in producing lower aliphatic
carboxylic acid ester, catalyst manufacture process and
method of use
IN Kadowaki, Etsuko; Higashi, Tomoyoshi; Oguchi, Wataru; Uchida, Hiroshi;
Narumi, Kousuke
PA Showa Denko K.K., Japan
SO U.S. Pat. Appl. Publ., 27 pp., Cont.-in-part of U.S. Ser. No. 70,259,
abandoned.
CODEN: USXXCO
DT Patent
LA English
FAN.CNT 3

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004242918	A1	20041202	US 2004-785229	20040225
JP 2002079090	A2	20020319	JP 2001-173624	20010608
WO 2002000589	A2	20020103	WO 2001-JP5532	20010627
WO 2002000589	A3	20020418		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, BG, BR, BY, BZ, CA, CH, CN,				

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, BG, BR, BY, BZ, CA, CH, CN,
DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
BJ, CF, CG, CI, CM, GA, GW, ML, MR, NE, SN, TD, TG 20010717
US 2003032837 A1 20030213 US 2001-889354 20011207
JP 2002316048 A2 20021029 JP 2001-373675 20020212
WO 2002064541 A1 20020822 WO 2002-JP1156 20020212
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
GM, GR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT,
LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NZ, OM, PH, PL, PT,
RO, RU, SD, SE, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG,
US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, BG, BR, BY, BZ, CA, CH, CN,
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, BG, BR, BY, BZ, CA, CH, CN,
CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
BF, BJ, CF, CG, CI, CM, GA, GW, ML, MR, NE, SN, TD, TG 20020304
US 2003118497 A1 20030626 US 2002-70259 20021203
ZA 2002009804 A 20030819 ZA 2002-9804 20030702
PRAI JP 2000-192964 A 20040312 ZA 2003-5143 20030702
US 2000-218803P P 20000718
JP 2001-36038 A 20010213
US 2001-273343P P 20010306
WO 2001-JP5532 W 20010627
US 2001-889354 B2 20010717
JP 2001-373675 A 20011207
WO 2002-JP1156 W 20020212
US 2002-70259 B2 20020304
CASREACT 142:40422

=> L2 NOT L3
L2 IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> S L2 NOT L3
L5 52 L2 NOT L3
=> LOG HOLD
COST IN U.S. DOLLARS
FULL ESTIMATED COST
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)
CA SUBSCRIBER PRICE
SESSION WILL BE HELD FOR 60 MINUTES
STN INTERNATIONAL SESSION SUSPENDED AT 09:22:59 ON 22 MAR 2006
Connecting via Winsock to STN

Since File Entry 92.05
Total Session 92.89
Since File Entry -3.75
Total Session -3.75

Welcome to STN International! Enter x:x
LOGINID:SSSPTA1623ZCT

PASSWORD:
***** RECONNECTED TO STN INTERNATIONAL *****
SESSION RESUMED IN FILE 'CAPLUS' AT 09:35:56 ON 22 MAR 2006
FILE 'CAPLUS' ENTERED AT 09:35:56 ON 22 MAR 2006
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)

L5 ANSWER 2 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2005:238937 CAPLUS
DOCUMENT NUMBER: 142:316483
TITLE: Preparation of lower aliphatic esters by vapor phase reaction of lower olefins with lower aliphatic carboxylic acids in the presence of a heteropoly acid catalyst.

INVENTOR(S): Fullerton, William
PATENT ASSIGNEE(S): BP Chemicals Limited, UK
SOURCE: PCT Int. Appl., 22 pp.
CODEN: PIXXD2

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005023747	A1	20050317	WO 2004-GB3619	20040824
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GE, GH, GM, GR, GU, HU, ID, IL, IN, IS, JP, KE, KG, KP, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MM, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, BG, GR, GU, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GN, GW, ML, MR, NE, SN, TD, TG				

PRIORITY APPLN. INFO.: CASREACT 142:316483 GB 2003-20692 A 20030903
OTHER SOURCE(S):
AB A process for making lower aliphatic esters comprises reaction of lower olefins with saturated lower aliphatic monocarboxylic acids in the vapor phase using heteropoly acid catalysts at 1200-1800 KPa. In a preparation of EtOAc a steam comprising ethylene 23.81 g/h, HOAc 3.65 mL/h, H₂O 1 mL/h, and Et₂O 0.54 mL/h at 10 barg and gas hour space velocity of 3600 was fed to a reactor packed with silicotungstic acid on silica at 185° to give, after 132 h on stream, byproduct acetaldehyde 0.14 g/1cat/h and MeCOEt 0.007 g/1cat/h.
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 3 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2004:756672 CAPLUS
DOCUMENT NUMBER: 141:279428
TITLE: Processes for the production of alkenyl esters of lower carboxylic acids and process for the production of alkenyl alcohols

INVENTOR(S): Saihata, Meiko; Uchida, Hiroshi
PATENT ASSIGNEE(S): Showa Denko K.K., Japan
SOURCE: PCT Int. Appl., 35 pp.
CODEN: PIXXD2

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004078698	A1	20040916	WO 2004-JP2216	20040225
W: AE, AE, AG, AL, AL, AM, AM, AT, AT, AU, AZ, AZ, BA, BB, BG, BG, BR, BR, BY, BY, BZ, BZ, CA, CH, CN, CO, CO, CR, CR, CU, CU, CZ, CZ, DE, DE, DK, DK, DM, DM, EC, EC, EG, EG, ES, ES, FI, FI, GB, GB, GE, GE, GM, GM, GR, GR, GU, GU, HU, HU, ID, ID, IN, IN				

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FILE 'CAPLUS' ENTERED AT 09:12:50 ON 22 MAR 2006
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L2 53 S ESTER AND (CARRIER OR SUPPORT OR SUPPORTING OR SUPPORTED) AND
L3 1 S L2 AND (SILICOTUNGSTIC OR PHOSPHOTUNGSTIC OR PHOSPHOLYBIC
L4 1 S L2 AND HETEROPOLYACID
L5 52 S L2 NOT L3

=> D 1-52 IBIB ABS

L5 ANSWER 1 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2005:347066 CAPLUS
DOCUMENT NUMBER: 142:392843
TITLE: Ziegler-Natta catalyst composition for ethylene polymerization

INVENTOR(S): Campbell, Richard E., Jr.; Chen, Linfeng; Painter, Roger B.; Reib, Robert N.; Tilston, Michael W.
PATENT ASSIGNEE(S): Dow Global Technologies Inc., USA
SOURCE: PCT Int. Appl., 18 pp.
CODEN: PIXXD2

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005035597	A1	20050421	WO 2004-US26641	20040818
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GE, GH, GM, GR, GU, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SI, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MM, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, BG, GR, GU, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GN, GW, ML, MR, NE, SN, TD, TG				

PRIORITY APPLN. INFO.: US 2003-505330P P 20030923
AB A process for the polymerization of olefin monomers comprises the steps of: contacting ethylene or a mixture of ethylene and one or more C4-8 α olefins with a catalyst composition comprising one or more Group 3-10 transition metal containing, Ziegler-Natta, procatalyst compds. (e.g., MgCl₂-supported TiCl₄); one or more alkylaluminum cocatalysts (e.g., triisobutylaluminum); and one or more polymerization control agents (e.g., Et p-ethoxybenzoate). The process is characterized in that at least one, such polymerization control agent is an alkyl or aryl ester of an aliphatic or aromatic (poly) carboxylic acid optionally containing one or more substituents comprising a Group 13, 14, 15, or 16 heteroatom.
REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GE, GM, KE, MM, MZ, SI, SZ, TZ, UG, ZM, AT, BE, CH, CY,
DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
BJ, CF, CG, CI, CM, GN, GW, ML, MR, NE, SN, TD, TG
AU 2002013766 A5 20020408 AU 2002-13766 20010822
US 2002065378 A1 20020530 US 2001-935333 20010822
US 6642325 B2 20031104 CN 2000-123560 A 20000822
WO 2001-CN1264 W 20010822

OTHER SOURCE(S): MARPAT 138:304690
AB The catalyst component contains a Ti-containing active component carried on a porous inert carrier, where the Ti-containing active component is a reaction product of at least one type of Ti compound (such as TiCl₄) with at least one type of Mg compound (such as MgCl₂) and at least one type of electron donor compound (such as Et acetate) in a molar ratio of 1:0.5-50:0.5-50, and may also contain at least one type of halide modifier (such as Cl₃CCl₂OH) (at a molar ratio of Ti compound/halide modifier = 1:0.5-50), and the porous inert carrier is spherical or spherical-like silica gel having average particle size 10-100 μm and sp. surface area 300-1000 m²/g. The catalyst component is combined with an alkyl aluminum compound (such as tri-Et aluminum) and used in gaseous or slurry polymerization or copolym. of ethylene, and especially used in preparing high-quality LLDPE resin by gaseous fluidized bed condensation technique.

L5 ANSWER 7 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2002:736941 CAPLUS
DOCUMENT NUMBER: 137:249510
TITLE: Preparation of a saturated carboxylic acid ester and use of water-soluble palladium complex

INVENTOR(S): Seavard, Jayasree; Seavard, Abdul Majeed; Sarkar, Bibhas Ranjan; Chaudhari, Raghunath Vitthal

PATENT ASSIGNEE(S): Council of Scientific & Industrial Research, India

SOURCE: U.S. Pat. Appl. Publ., 9 pp.
CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
US 2002137964 A1 20020926 US 2001-813086 20010320
US 6479693 B2 20021112 US 2001-813086 20010320

PRIORITY APPL. INFO.: MARPAT 137:249510

OTHER SOURCE(S):
AB A carboxylic acid ester R₃R₄SCC(R₁R₂)CO₂R (I) is prepared, where R = alkyl or aryl, R₁ = aryl, substituted aryl, naphthyl or substituted naphthyl or alkyl, R₂-5 = H or alkyl, by reacting an olefin R₃R₄SCC(R₁R₂) (R₁ = aryl, substituted aryl, naphthyl or substituted naphthyl or alkyl, R₂-4 = H or alkyl), in the presence of an alc. and an organic solvent and a supported aqueous phase Pd complex catalyst, and in presence or absence of a protonic acid and an alkali metal halide, under CO atmospheric, cooling the reaction mixture to ambient temperature, depressuring the reactor, flushing the reaction vessel with inert gas, separating the catalyst by filtration, and removing the solvent and isolating I. Thus, styrene (0.144 mol), MeOH, cyclohexane, and Pd(TPPTS)₃ [TPPTS = tris(sodium 3-sulfonatophenyl)phosphine] was heated to 75°, pressurized to 500 psig with CO, and stirred 12 h to give a mixture of 2- and 3-methylphenyl propionates, selectivity 54.26% and 45.73%, resp.

L5 ANSWER 8 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

2002:637632 CAPLUS
137:171395
Process for the use of and supports for the manufacture of catalysts for producing lower aliphatic carboxylic acid esters via the addition-esterification reaction of lower aliphatic carboxylic acids with lower alkenes

INVENTOR(S): Kadowaki, Etsuko; Narumi, Kousuke; Uchida, Hiroshi
PATENT ASSIGNEE(S): Showa Denko K. K., Japan
SOURCE: PCT Int. Appl., 45 pp.
CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 3

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
WO 2002064541 A1 20020822 WO 2002-JP1156 20020212
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NZ, OM, PA, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, BG, BZ, MD, RU, TJ, TM, RW: GH, GM, KE, LS, MM, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE, SN, TD, TG
JP 2002316048 A2 20021029 JP 2001-373675 20011207
EP 1360166 A1 20031112 EP 2002-711471 20020212
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BR 2002007225 2002-7225 20020212
CN 1491204 A 20040309 CN 2002-804859 20020212
US 2003118497 A1 20040421 CN 2002-804859 20020304
ZA 2003005143 20040312 ZA 2003-5143 20030702
US 2004242918 A1 20041202 US 2004-785229 20040225
PRIORITY APPL. INFO.: JP 2001-36038 A 20010306
JP 2001-373675 A 20011207
JP 2000-192964 A 20000627
JP 2000-218803P P 20000718
WO 2001-JP5532 W 20010627
US 2001-889354 B2 20010717
WO 2002-JP1156 W 20020212
US 2002-70259 B2 20020304

AB A siliceous support for use in a catalyst for producing a lower aliphatic carboxylic acid esters (e.g., Et acetate) by the esterification-addition reaction of a lower olefin (e.g., ethylene) with a lower aliphatic carboxylic acid (e.g., acetic acid) in the gas phase is described and the support has a silicon content of 39.7-46.3% by mass or a silicon content of from 85-99% by mass in terms of silicon dioxide or a crush strength of 230 N; the catalyst is a heteropoly acid or salt on the support. By the use of a catalyst comprising the support, a lower aliphatic carboxylic acid ester is produced from a lower olefin and a lower aliphatic carboxylic acid without causing a great reduction of catalytic activity or cracking or abrasion of the catalyst.

REFERENCE COUNT: 4
THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 9 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2000:881210 CAPLUS
DOCUMENT NUMBER: 134:42585

TITLE:

Process for producing ethylene-olefin
interpolymersFord, Randal Ray; Vanderbilt, Jeffrey James; Williams,
Darryl Stephen

Eastman Chemical Co., USA

PCI Int. Appl., 64 pp.

CODEN: PIXXDZ

Patent

English

1

PARENT ASSIGNEE(S):

SOURCE:

DOCUMENT TYPE:

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PARENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2000075202	A1	20001214	WO 1999-US22957	19991014
US 6417301	B1	20020709	US 1999-386545	19990831
CA 2375621	AA	20001214	CA 1999-2375621	19991014
BR 9917354	A	20020226	BR 1999-17354	19991014
EP 1183284	A1	20020306	EP 1999-351735	19991014
EP 1183284	B1	20050427		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
JP 200301526	T2	20030114	JP 2001-502482	19991014

PRIORITY APPLN. INFO.:

AB The process uses Ziegler-Natta catalysts for producing ethylene-olefin interpolymers having a given melt index and d. and a reduced melting peak temperature (Tm). The process involves contacting ethylene and 21 other olefin under polymerization conditions with a Ziegler-Natta catalyst and 21 modifier comprising 21 element from Group 15 and/or Group 16 in amts. sufficient to reduce the melting peak temperature of the interpolymers.

THERE ARE 5 CITED REFERENCES AVAILABLE IN THE RE FORMAT

REFERENCE COUNT:

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 10 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

DOCUMENT NUMBER:

TITLE:

INVENTOR(S):

PARENT ASSIGNEE(S):

SOURCE:

DOCUMENT TYPE:

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PARENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5928980	A	19990727	US 1997-795669	19970206
US 5928980	A	19990727	US 1997-795669	19970206

AB A heavy metal poisoned, spent FCC catalyst is treated by chemical impregnating the poisoned catalyst with a new catalytic metal or metal salt to provide an attrition resistant catalyst or sorbent for a different catalytic or absorption processes, such as catalysts for Fischer-Tropsch synthesis, and sorbents for removal of sulfur gases from fuel gases and flue gases. The heavy metal contaminated FCC catalyst is directly used as a support for preparing catalysts

having new catalytic properties and sorbents having new sorbent properties, without removing or passivating the heavy metals on the spent FCC catalyst as an intermediate step.

THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

REFERENCE COUNT:

L5 ANSWER 11 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

DOCUMENT NUMBER:

TITLE:

A process for the preparation of a magnesium halide-

supported metallocene polymerization catalyst

Sensarma, Soumen; Sivaram, Swaminathan

Council of Scientific and Industrial Research, India

Eur. Pat. Appl., 10 pp.

CODEN: EPXNDW

Patent

English

DOCUMENT TYPE:

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PARENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 878484	A1	19981118	EP 1997-303286	19970514
EP 878484	B1	20041215		

R: BE, CH, DE, FR, GB, LI

PRIORITY APPLN. INFO.:

IN 1997-DE795 A 19970327

AB A magnesium halide-supported metallocene catalyst, useful for

polymerization of olefins in slurry or gas phase, is manufactured

by (a) preparing a solution (solution A) of the magnesium halide compound via

Grignard decomposition which consists of preparing the slurry of magnesium

metal

in an electron donor solvent, and heating the slurry of magnesium metal to

0°C to 50°C for a period of 10 min to 4 h while adding a

dihaloalkane compound; (b) sep. preparing a solution of metallocene compound

into the same electron donor solvent (solution B); (c) heating the solution B to

0°C to 50°C for a period ranging between 10 min to 1 h; (d)

adding solution B into solution A in a period ranging between 10 min to 2 h,

while keeping the temperature in the range of 0 to 50°C; (e) cooling the

resultant homogeneous solution to room temperature under inert atmospheric;

(f) pouring

the reaction mixture into a liquid hydrocarbon medium in which all the

components will be partly or completely insol. to precipitate the catalyst; (g)

separating the solid catalyst precipitated by conventional methods; (h)

washing the

solid catalyst by a hydrocarbon solvent; and (i) drying the solid under

vacuum at a temperature ranging between 0 to 50°C.

THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

REFERENCE COUNT:

L5 ANSWER 12 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

DOCUMENT NUMBER:

TITLE:

Catalyst and its use in the preparation of unsaturated

carboxylic acid esters

White, James F.; Slawski, Barbara; White, Geoffrey

Engelhard Corp., USA

U.S., 11 pp.

CODEN: USXXAM

Patent

English

DOCUMENT TYPE:

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PARENT INFORMATION:

PATENT NO.

KIND

DATE

APPLICATION NO.

DATE

US 5347046 A 19940913 US 1993-67445 A 19930525
CA 2161952 AA 1941208 CA 1994-2161952 19940511
WO 9427720 CN 1941208 WO 1994-US5382 19940511
R: AU, BR, CA, CN, JP, KR, RU, GB, GR, IE, IT, LU, MC, NL, PT, SE
RW: AT, BE, CH, DE, DK, ES, FR, AU 1994-69129 19940511
AU 9406996 A 19960319 BR 1994-6696 19940511
BR 9406996 A 19960319 EP 1994-917387 19940511
EP 708684 A 19960501 EP 1994-917387 19940511
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE
CN 1123527 A 19960529 CN 1994-192124 19940511
JP 08510685 T2 19961112 JP 1995-500722 19940511
JP 3698322 B2 20050921

PRIORITY APPLN. INFO.:

US 1993-67445 A 19930525
WO 1994-US5382 W 19940511
AB Title catalysts comprise (i) ≥ 1 palladium group metals and/or
comps. thereof; (ii) gold and/or comps. thereof; and (iii) optionally,
 ≥ 1 promoters selected from Cu, Ni, Co, Fe, Mn, Pb, Ag and their
comps. Preferably these catalyst comps. contain ≥ 1 promoters
wherein the gram ratio of metal in the promoter to palladium group metal
is ≤ 0.4 , and the catalysts are deposited on a support
material treated with ≥ 1 alkali metal bases and ≥ 1 alkali
metal silicates prior to deposition. In some cases, the catalysts are
further impregnated with ≥ 1 alkali or alkaline-earth carboxylates as
promoters and activators. Ethylenically unsatd. esters are
prepared with improved activity and selectivity by reacting in the
vapor phase ≥ 1 olefinic comps. with ≥ 1
carboxylic acid and mol. oxygen in the presence of the foregoing
catalyst comps. Thus, vapor-phase reaction of ethylene at
140° with HOAc and O over silica beads that were pretreated with
KOH and K silicate solution at 16.2 and 127.2 g/L beads, resp., and
impregnated with Na2PdCl4, NaAuCl4, CuCl2, and KOAc to contain 4.1, 1.8,
6.3, and 22 g/L catalyst of Pd, Au, Cu, and KOAc, resp., gave vinyl
acetate at space-time yield 158.3 (93% selectivity) and selectivity 96.3%
(12% space-time yield).

L5 ANSWER 13 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1994:656552 CAPLUS

DOCUMENT NUMBER: 121:256552

TITLE: Components and catalysts for the polymerization of

olefins.

INVENTOR(S): Sacchetti, Mario; Cuffiani, Illaro; Pennini, Gianni

PATENT ASSIGNEE(S): Spherilene S.r.l., Italy

SOURCE: Eur. Pat. Appl., 15 pp.

CODEN: EPAXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 601525	A1	19900210	EP 1993-119646	19931206
EP 601525	B1	19900210		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, NL, PT, SE				
IN 181561	A	19980711	IN 1993-CA721	19931124
AT 176676	E	19990215	AT 1993-119646	19931206
ES 2129063	T3	19990601	ES 1993-119646	19931206
AU 9352295	A1	19940623	AU 1993-52295	19931208
AU 673551	B2	19961114		
ZA 9309210	A	19940808	ZA 1993-9210	19931208
JP 07306507	A2	19951114	JP 1993-308020	19931208
RU 2126420	C1	19990220	RU 1993-54328	19931208
NO 9304496	A	19940613	NO 1993-4496	19931209
IL 107958	A1	19980924	IL 1993-107958	19931209

FI 9305550 A 19940612 FI 1993-5550 19931210
FI 112240 B1 20031114
BR 9305019 A 19940705 19931210
CN 1089622 A 19940720 19931211
CN 1034580 B 19970415
CA 2111308 AA 19940612 19931213
CN 1127262 A 19960724 19950918
CN 1041525 B 19990106
US 2001011060 A1 20010802
US 6388028 B2 20020514
US 1997-878681 19970619
IT 1992-MI2822 A 19921211
US 1993-163571 B1 19931208
US 1996-741580 B1 19961101

PRIORITY APPLN. INFO.:

AB The present invention relates to spherical solid catalyst components for
the polymerization of olefins, comprising a titanium compound,
supported on a magnesium halide, containing more than one Ti-halogen
link and optionally containing groups different from halogen in an amount lower
than 0.5 mol per mol of Ti. Spherical solid comps. of the invention are
characterized by having a surface area, measured by the BET method, of
lower than 70 m²/g, a total porosity, measured by the mercurium method,
higher than 0.5 cm³/g and a pore radius such that at least 50% have values
higher than 800 Å. The polyolefins prepared by vapor
-phase-polymerization in the presence of these catalysts have spheroidal
particles with high bulk d. Thus, vapor-phase-polymerization of
ethylene in the presence of (iso-Bu)3Al and spheres with overall Ti
content 6%, Ti3+ content 4.9, Al content 3%, Mg content 12.2%, Cl content
68.2%, OEt content 0.3%, BET porosity 0.208 cm³/g (of which 50% was due to
pores with radius >300 Å), BET surface area 56.2 m²/g, mercurium total
porosity 0.674 cm³/g (of which 50% was due to pores with radius >1250
Å, 91% of the pores have a radius of $\leq 15,000$ Å) and
mercurium surface area 21 m²/g at 75° gave polymer powder having
spheroidal particles with bulk d. 0.32 g/cm³.

L5 ANSWER 14 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1994:322766 CAPLUS

DOCUMENT NUMBER: 120:322766

TITLE: Preparation of unsaturated carboxylic acid

esters with palladium catalysts

INVENTOR(S): Harada, Hirobumi; Hamachi, Hideyuki; Nishizuka,

Masaya; Kato, Takao; Saito, Toshihiro

PATENT ASSIGNEE(S): Tosoh Corp, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06025082	A2	19940201	JP 1992-200102	19920706
OTHER APPLN. INFO.:			JP 1992-200102	19920706

AB Unstd. carboxylic acid esters are prepared by reaction

of olefins, O, and ACOH in gas phases in presence of

Pd catalysts prepared by ion exchange of OH on the surfaces of catalyst

supports with Pd amine complexes, optional calcination under

O-containing gases, reduction of the supports, and

supporting alkali metal acetates on the supports.

Silica was impregnated with an aqueous NH3 solution of (Pd(NH3)4Cl2), dried at

110° for 3 h, heated at 400° for 5 h, reduced by H at

300° for 5 h, impregnated with aqueous ACOH, and dried at 110°

for 3 h to prepare a Pd- and ACOH-containing catalyst. A 50:9:7:33 mixture of

propylene, O, ACOH, and N was passed through the catalyst at 150°

5 atm, and 13.6 NL/h to give 307 g/L/h allyl acetate with 95.8%

selectivity.

L5 ANSWER 15 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1994:191137 CAPLUS
DOCUMENT NUMBER: 120:191137

TITLE: Preparation of lower fatty acid **esters**
INVENTOR(S): Suzuki, Toshio; Takahashi, Shunel; Sano, Kenichi
PATENT ASSIGNEE(S): Showa Denko KK, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKKXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05294894	A2	19931109	JP 1992-101364	19920421
JP 2850636	B2	19990127		
IN 189050	A	20021214	IN 2000-DE957	20001025
			JP 1992-101364	A 19920421

PRIORITY APPLN. INFO.: CASREACT 120:191137

AB The title compds. are prepared by **gas-phase esterification** of lower fatty acids with lower **olefins** using **supported** catalysts of heteropoly acid salts of Li, Cu, Mg, and/or Ca. A reactor containing Li phosphotungstate **supported** on SiO₂ (preparation given) was fed with mixed **gases** containing AcOH and CH₂:CH₂ at 150° and 5 kg/cm²-gauge to give 91.8% Et acetate.

L5 ANSWER 16 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:433032 CAPLUS
DOCUMENT NUMBER: 117:33032

TITLE: Steam stripping for removal of organic pollutants from water. 2. **Vapor-liquid** equilibrium data

AUTHOR(S): Hwang, Ying Long; Olson, James D.; Keller, George E., II

CORPORATE SOURCE: Union Carbide Chem. and Plast. Co. Inc., South Charleston, WV, 25303, USA

SOURCE: Industrial & Engineering Chemistry Research (1992), 31(7), 1759-68

CODEN: IECRED; ISSN: 0888-5885

DOCUMENT TYPE: Journal

LANGUAGE: English

AB In **support** of the simple tools for steam stripping presented in part 1, the estimation of the infinite-dilution **vapor-liquid** equilibrium consts. (K_∞) for organic pollutants in dilute aqueous solns are discussed. A databank of K_∞ at 1 atm and 100° as well as at 25° was established for a number of common organic pollutants. In addition, a correlation

of K_∞ with mol. structure was formulated. The concept that the effectiveness of stripping an organic pollutant from water is mainly

determined by its hydrophobicity (indicated by its low aqueous solubility) rather than by its pure-component volatility is also clarified. On the basis of the guideline proposed in part 1, the estimated K_∞ values reveal that steam stripping is effective in removing hydrophobic (sparingly soluble) pollutants such as paraffins, **olefins**, aroms., halogenated hydrocarbons, and compds. with dominant hydrocarbon segments.

L5 ANSWER 17 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:237786 CAPLUS
DOCUMENT NUMBER: 116:237786

TITLE: Hollow cylindrical **carrier** as catalyst **support** for production of unsaturated **esters**

INVENTOR(S):
PATENT ASSIGNEE(S):
SOURCE:

Matsumoto, Kenji; Fuchigami, Yoshio
Kuraray Co., Ltd., Japan
Eur. Pat. Appl., 16 pp.

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 464633	A1	19920108	EP 1991-110562	19910626
EP 464633	B1	19940119		
	R: BE, DE, FR, GB, IT			
JP 04227069	A2	19920817	JP 1991-99591	19910403
US 5371277	A	19941206	US 1994-227700	19940414
			JP 1990-177125	A 19900703
			US 1991-723376	B1 19910628
			US 1993-52292	B1 19930426

PRIORITY APPLN. INFO.:

AB Production of unsatd. **esters** with high catalyst activity and low pressure drop in the catalyst layer is achieved by **gas-phase** reaction of **olefin, carboxylic acid**, and O in the presence of a catalyst (e.g. Pd) **supported** on a hollow cylindrical **carrier** having λ l through channel. A **gas** containing C₂H₄ 72, O 8, and AcOH 20 volume% was passed through a catalyst layer (containing Pd 5.0, Au 0.5, and KOAc 20 g/L on a hollow cylindrical silica) at 160° and 2000 h-1 giving vinyl acetate with selectivity 94% and pressure drop 0.20 kg/cm², vs. 95 and 0.26 resp., when using a spherical silica **support**.

L5 ANSWER 18 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:227394 CAPLUS
DOCUMENT NUMBER: 116:227394

TITLE: Chromatographic method for analysis of aliphatic

olefin alcohols, acetates and ketones

INVENTOR(S): Nesterova, I. P.; Rastegaeva, V. M.

PATENT ASSIGNEE(S): All-Union Scientific-Research Institute of Biological Methods of Plant Protection, USSR

SOURCE: U.S.S.R. From: Otkrytiya, Izobret. 1991, (31), 182.

CODEN: URXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Russian

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SU 1672352	A1	19910823	SU 1988-4445933	19880513
			SU 1988-4445933	19880513

PRIORITY APPLN. INFO.:

AB The mixture is separated in a capillary column with a stationary phase in a **carrier-gas** flow with subsequent detection of separated **isomers**. To increase separation selectivity and expand the circle of sample substances - among them insect sex attractants, liquid-crystal cholesteryl-p-methoxybenzoate is used as the stationary phase, and the separation is performed at 170-190° for a **carrier-gas** rate in the column over the interval of 6.0-15.0 cm/s.

L5 ANSWER 19 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:173577 CAPLUS
DOCUMENT NUMBER: 116:173577

TITLE: Preparation of catalysts for unsaturated **ester**

INVENTOR(S): manufacture

Kumai, Toshi; Takagi, Isato; Morofuji, Masamitsu;

Fujiwara, Hideatsu; Yoshida, Soichi

PATENT ASSIGNEE(S): Tosoh Corp., Japan; Japan Synthetic Rubber Co., Ltd.

SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

Patent

Japanese

1

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

JP 03275141 A2 19911205 JP 1990-77211 19900327

PRIORITY APPLN. INFO.: JP 1990-77211 19900327

OTHER SOURCE(S): CASREACT 116:173577

AB Pd catalysts for preparation of unsatd. diesters from olefins,

carboxylic acids, and O are prepared by treating metal compds. and

supports in aqueous tartaric acid containing 0.1-10% hydrogen halides and

subsequent reduction. A mixture of PdCl₂ 13.4, Sb₂O₃ 21.9, and NH₄VO₃ 8.8 g was

dissolved in an aqueous solution containing 25% tartaric acid and 2.5% HCl,

then 150

g silica gel was stirred in the solution, dried, calcined at 400° for

20 h, and reduced in H at 200° for 2 h and at 400° for 2 h.

The solid was then impregnated in an aqueous solution containing 24 g CsCl and

24 g

CsOAc and dried to give a Pd catalyst. A mixed gas of

butadiene, AcOH, O₂, and N₂ (20/20/12/48) was treated in the presence of the

catalyst at 185° for 100 h to give 95.3% diacetylbene containing

95.1% 1,4-isomer.

L5 ANSWER 20 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

1991:494244 CAPLUS

DOCUMENT NUMBER:

115:94244

TITLE: Preparation and uses Ziegler-Natta polymerization

catalysts

INVENTOR(S): Baillly, Jean Claude Andre; Behue, Patrick

BP Chemicals Ltd., UK; BP Chemicals SNC

Source: Eur. Pat. Appl., 11 pp.

CODEN: EPXDXM

Patent

English

1

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

EP 437080 A1 19910717 EP 1990-314081 19901220

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE

FR 2656615 B1 19930507 FR 1990-191 19900104

US 5106805 A 19920421 AU 1990-68355 19901217

AU 9068355 A1 19910711 AU 1990-77211 19901220

CA 2033028 AA 19910705 CA 1990-2033028 19901221

NO 9005634 A 19910705 NO 1990-5634 19901228

JP 04039309 A 19920210 JP 1990-408659 19901228

FI 9100020 A 19910705 FI 1991-20 19910102

HU 59702 A2 19920629 HU 1991-17 19910103

CN 1053243 A 19910724 CN 1991-100033 19910104

BR 9100019 A 19911022 BR 1991-19 19910104

FR 1990-191 A 19900104

PRIORITY APPLN. INFO.: Ziegler-Natta catalysts, useful for the polymerization of olefins and

the manufacture of elastomers, are prepared by contacting with a liquid

hydrocarbon as spherical support comprising MgCl₂ and an

electron donor compound free of labile H, successively with an electron

donor compound containing labile H, and then with an organometallic compound

capable of reducing V and Ti compds., washing the solid product with a

liquid hydrocarbon, and then contacting the washed solid product with a

hydrocarbon-soluble V and Ta compds. Thus, spherical particles of MgCl₂

support (prepared by reacting Bu₂Mg with tert BuCl and diisomyl ether) in C₆H₁₄ was heated with Et₃Al, VOCI₃ and TiCl₄ to give as spherical catalyst having the following molar ratio: V/Mg = 0.15, Ti/Mg = 0.04, Al/Mg = 0.15, and Cl/Mg = 2.6. The catalyst was used for the gas -phase copolymn. of ethylene and propylene.

L5 ANSWER 21 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

1991:187321 CAPLUS

DOCUMENT NUMBER:

114:187321

TITLE: Preparation of elastomeric propylene copolymers by

gas-phase polymerization in presence of

Ziegler-Natta catalysts

INVENTOR(S): Baillly, Jean Claude Andre; Bordere, Louis

BP Chemicals Ltd., UK

Source: Eur. Pat. Appl., 14 pp.

CODEN: EPXDXM

Patent

English

1

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

EP 415588 A1 19910306 EP 1990-308836 19900810

R: AT, BE, CH, DE, DK, ES, GB, GR, IT, LI, LU, NL, SE

FR 2651234 A1 19910301 FR 1989-11523 19890829

AT 2651234 B1 19930312 AT 1990-308836 19900810

ES 1990-308836 T3 19941216 ES 1990-DE820 19900816

IN 179795 A 19971213 CA 1990-2023653 19900820

CA 2023653 AA 19910301 AU 1990-61330 19900824

AU 9061330 A1 19910307 DD 19920409 19900827

AU 622573 B2 19920409 DD 1990-343664 19900828

DD 298936 A5 19920319 NO 1990-3763 19900828

NO 9003763 B 19940613 HU 1990-5509 19900828

NO 175263 C 19940921 BR 1990-4245 19900828

HU 54715 A2 19910328 CN 1990-107047 19900829

JP 03093803 A2 19910418 EP 1989-11523 A 19890828

BR 9004245 A 19910903 EP 1990-308836 A 19900810

CN 1050199 A 19910327

PRIORITY APPLN. INFO.: AB Elastomeric copolymers of 20-80% propylene (I) with 80-20% ethylene (II) and/or 1-butene (III) and optionally, ≥ 1 C₅-12 α -olefins are prepared by a 2-stage procedure, prepolymerizing in the first stage I, optionally mixed with II and/or C₄-12- α olefin, in the presence of a Ziegler-Natta catalyst and a cocatalyst consisting of a trialkylaluminum and/or in alkyl aluminum halide not complexed with an external electron donor compound, followed by a gas-phase copolymn. of the prepolymer with a mixture of I, II, and/or III and optionally a C₅-12- α olefin in a proportion so that I represents 20-80 volume% of the copolymerizable olefins. The Ziegler-Natta catalyst is prepared by contacting MgCl₂ support with an internal electron donor compound, e.g., aromatic carboxylic acid esters, and then with TiCl₄, provided the support is initially preactivated by the different organic electron donor compound which is free from labile H, and provided the support consists of 80-99.5 mol% MgCl₂ and 0.5-20 mol% electron donor compound. Thus, a preactivated support comprises MgCl₂ in hexane (IV) was successively stirred with BuOH, PhCO₂Et, and TiCl₄, with intermediate decanting, washing, and resuspension in IV to give a particulate catalyst containing 6.5% Ti atoms per Mg atom and having a specific particle

size distribution. I was then prepolymerized in the presence of the catalyst and Et₂AlCl in IV to give a prepolymer suspension in IV. The prepolymer was then copolymerized in a fluidized bed reactor with I and II in the presence of anhydrous II-I copolymer and (iso-Bu)₃Al to give an agglomerated II-I copolymer containing 63% II and having a bulk d. 0.45 g/cm³.

L5 ANSWER 22 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1985:423053 CAPLUS
DOCUMENT NUMBER: 10323053

TITLE: Deodorization of polyolefins
INVENTOR(S): Ube Industries, Ltd., Japan
PATENT ASSIGNEE(S): Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKKXAF

DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 60038408	A2	19850228	JP 1983-146694	19830812
JP 04036164	B4	19920615		

PRIORITY APPLN. INFO.:

AB Polyolefins prepared by polymerizing C₂₃ α-olefins in the presence of a solid catalyst component containing Mg, halogen, and Ti, an organoaluminum compound, and an aromatic carboxylic acid ester were deodorized by contacting with C₂₈ alc.

vapor. Thus, 15 mmol suspended in 30 mL toluene was treated over 30 min with a solution of 15 mmol MeSi(OEt)₃ in 10 mL toluene at room temperature,

heated at 60° for 1 h, cooled to -10°, treated over 30 min with a solution of 27 mmol BuMgCl in diisopropyl ether, and heated at 30° for 1 h to obtain a carrier. The carrier

suspended in 30 mL toluene was heated with 150 mmol TiCl₄ and 6.5 mmol EtOBz at 90° for 1 h and filtered hot to obtain a solid product which was then suspended in toluene heated with 150 mmol TiCl₄ at

90° for 1 h, and filtered hot to obtain a solid catalyst component (I) with Ti content 2.8%. A heptane suspension of 16 mg I, 0.24 mmol Me₂OC₆H₄Me-p in 10 mL heptane, and 0.94 mmol Et₃Al in 4 mL heptane were

mixed and autoclaved with 1200 mL liquid propylene at 65° for 1 h to obtain 250 g powdered polypropylene [9003-07-0] with boiling heptane-insol. content 94.5%. The polymer (200 g) fluidized with N was contacted with

120 g/h 2-propanol [67-63-0] vapor at 110° for 30 min to obtain substantial deodorization.

L5 ANSWER 23 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1980:41339 CAPLUS
DOCUMENT NUMBER: 92:41339

TITLE: Supported catalyst for producing unsaturated

esters from C₃-C₁₀ olefins, carboxylic acids, and oxygen in the gas phase

INVENTOR(S): Wunder, Friedrich A.; Quadflieg, Therese; Roscher, Guenter; Heck, Guenter

PATENT ASSIGNEE(S): Hoechst A.-G.; Fed. Rep. Ger.
SOURCE: Ger. Offen., 19 pp.
CODEN: GWXXBX

DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2811211	A1	19790927	DE 1978-2811211	19780315

PRIORITY APPLN. INFO.:

AB The efficiency of catalysts for the title reaction was improved by using catalyst supports consisting of particles with star-shaped cross-sections or ribbed particles (vs. pellets or Raschig rings). Thus, 1377 g SiO₂ particles (5-pointed star cross-section), point ht. 1.7 mm, diameter 6 mm, bulk d. 0.27 kg/L were treated with 63 g Pd(OAc)₂, 63 g KOAc, and 50 g Bi(OAc)₃ in 1093 mL HOAc and 13 g Ba aurate and 84 g H₂O in 393 mL HOAc and the catalyst was dried at 60/270 mbar under N. The catalyst gave 963 g/L h conversion of propene and HOAc to allyl acetate and 648 g/L h conversion of isobutene and HOAc to methylallyl acetate. Data for runs with 4 conventional supports were also given.

L5 ANSWER 24 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1978:406804 CAPLUS
DOCUMENT NUMBER: 89:6804

TITLE: Unsaturated esters
INVENTOR(S): Klass, Donald L.
PATENT ASSIGNEE(S): Union Oil Co., USA

SOURCE: U.S., 6 pp.
CODEN: USXXAM

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4057375	A	19771108	US 1976-699198	19760623
US 4161610	A	19790717	US 1961-141832	19610929

PRIORITY APPLN. INFO.:

AB Olefins are oxidized to unsatd. esters of organic acids or acetals and/or) unsatd. ethers of alcs. by contacting the olefin with an organic acid or alc. reactant in the presence of a catalyst comprising a compound of a group VIII noble metal and a redox agent; the catalyst is kept active by contacting it with O. Thus, C₂H₄ [74-85-1] vapor was passed through liquid HOAc [64-19-7] to vaporize some of the HOAc and the resulting mixture was passed through a catalyst bed consisting of PdCl₂, HCl, and CuCl₂ supported on silica. Vinyl acetate [108-05-4] was removed as product from the effluent gas stream, and the unreacted C₂H₄ was recycled to the reactor. The flow of C₂H₄ and HOAc to the reactor was interrupted periodically, and a stream of O was passed through the catalyst bed to regenerate the catalyst.

L5 ANSWER 25 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1977:161909 CAPLUS
DOCUMENT NUMBER: 86:161909

TITLE: Catalysts for organic acetate production

PATENT ASSIGNEE(S): Bayer A.-G.; Fed. Rep. Ger.
SOURCE: Jpn. Tokkyo Koho, 4 pp.
CODEN: JAXXAD

DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 51025438	B4	19760730	JP 1967-52769	19670818
CA 954529	A1	19740910	CA 1964-903693	19640527

BE 648814 A 19641204 BE 1964-648814 19640604
NL 6406336 A 19641207 NL 1964-6336 19640604

PRIORITY APPLN. INFO.: DE 1963-F39909 A 19630604
AB Pd salt supported by a catalytic carrier is reduced to

Pd metal in an aqueous alkaline solution containing reducing agent. The carrier is then impregnated with alkali and/or alkaline earth metal acetates and dried to give a catalyst for **gas**-phase production of organic acetates from a mixture of **olefin**, O, and HOAc. Thus, Al₂O₃ catalytic **carriers** (80 m²/g surface area) were impregnated with aqueous PdCl₂ solution, then immersed in an alkaline NaOH solution the **carriers** contained 2 weight % Pd at this stage. The **carriers** were then impregnated with aqueous solution containing LiOAc (12 weight parts/100 weight parts final catalyst) and dried at 150° under vacuum to give catalysts. A 500 cm³ catalyst sample was then placed in a 1500 mm long tube (22 mm inner diameter) and a mixture of ethylene 2.08, HOAc 1.83, and O 0.42 mol/hr was passed through the tube at 135° and 1 atm. The products of the ethylene was converted with 87.3% of the products as vinyl acetate and 12.7% of the products as CO₂.

L5 ANSWER 26 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1977:161905 CAPIUS
DOCUMENT NUMBER: 86:161905
TITLE: Silica catalytic **supports**
INVENTOR(S): Kawai, Kohichiro; Fujiwara, Yuzuru; Nakamura, Michihiro
PARENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
JP 52004490 A2 19770113 JP 1975-79804 19750627
PRIORITY APPL. INFO.: JP 1975-79804 A 19750627
AB Sintered silica having a surface area of 150-600 m²/g, total pore volume of 0.3-3.0 mL/g, volume of 40-60 Å diameter pores 0.1-1.2 mL/g, volume of 800-1300 Å diameter pores 0.1-0.5 mL/g, and bulk d. 0.2-0.55 g/mL is useful as catalyst **carrier**. The **supports** are especially useful for catalysts used for **gas** phase synthesis of unsatd. **esters** from **olefins**. Thus, Aerogel (from Fuji Davison Chemical Co.) was sintered 4 h at 900°, pulverized to give SiO₂ powder (4 µ average diameter, porosity 0.65 mL/g, surface area 210 m²/g, average micropore diameter 112 Å, bulk d. 0.50 g/mL), 5 kg of which was mixed with 9 L of aqueous 10 weight % Snowtex-N (silica gel from Nissan Kagaku Kogyo K.K.), and the mixture was made into 5-mm-diameter spherical pellets, the pellets were dried at 100° (5 h) and sintered at 700° (4 h) to give catalytic **support** whose surface area, total pore volume, small pore (40-60 Å) volume, large pore (1000-1300 Å) volume, bulk d., and compressive strength were 252 m²/g, 1.14, 0.6, 0.25 mL/g, 0.38 g/mL, and 14.0 kg/pellet, resp. The **support** was then impregnated with Pd 3.3, Au 1.5, and KOAc 30 g/L-silica, and vinyl acetate was synthesized from a mixture of ethylene 60, O 10, HOAc 30 mol. % at 145°, space velocity 800/h (at 0°, 1 atm), and at 1 atm: the rate of vinyl acetate production was 2.80 ton/m³-catalyst-day.

L5 ANSWER 27 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1975:605969 CAPIUS
DOCUMENT NUMBER: 83:205969
TITLE: Carboxylic acid **ester**
INVENTOR(S): Onoda, Takeru; Wada, Keisuke; Otake, Masayuki
PARENT ASSIGNEE(S): Mitsubishi Chemical Industries Co., Ltd., Japan
SOURCE: Ger. Offen., 22 pp.
CODEN: GWWXBX
DOCUMENT TYPE: Patent

LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
DE 2510089 A1 19750918 DE 1975-2510089 19750307
JP 50121191 A2 19750922 JP 1974-28416 19740312
JP 56034345 B4 19810810
JP 51008188 A2 19760122 JP 1974-79007 19740710
JP 56021463 B4 19810519 NL 1975-2572 19750305
NL 7502572 A 19750916 FR 1975-7540 19750311
FR 2263999 A1 19751010 GB 1975-10076 19750311
GB 1462749 A 19770126 JP 1974-28416 A 19740312
JP 1974-79007 A 19740710

AB Aromatic compds. or **olefins** were converted into **esters** by reaction with O and HOAc in the **gas** phase in the presence of a catalyst consisting of Pd, Sb and K or Zn acetate. The catalyst **support** (SiO₂) was first soaked in an aqueous solution of Sb compound (e.g., SbCl₃), dried, and calcined in a stream of O, then soaked in Pd salt and HOAc salt solution, dried and calcined. Reaction of C₆H₆ with O and HOAc in the presence of these catalysts gave PhOAc.

L5 ANSWER 28 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1975:98874 CAPIUS
DOCUMENT NUMBER: 82:98874

TITLE: Carbon-chain polymers
INVENTOR(S): Pomogailo, A. D.; Lisitskaya, A. P.; Ponomareva, T. I.; Matkovskii, P. E.; Raspopov, L. N.; Rozenberg, B. A.; D'yachkovskii, F. S.
PARENT ASSIGNEE(S): Institute of Chemical Physics, Chernogolovka, USSR
SOURCE: U.S.S.R. From: Otkrytiya, Izobret., Prom. Obraztsy, Tovarnye znaki 1974, 51(33), 74.
CODEN: URXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE
SU 442187 T 19740905 SU 1972-1852733 19721130
PRIORITY APPL. INFO.: SU 1972-1852733 A 19721130
AB In melt, solvent, or **gas** phase polymerization of α-**olefins**, optionally with dienes, in the presence of an organic metallic compound, polymer yield was improved when the catalyst **support** was hydrolyzed styrene-vinyl acetate copolymer [25213-29-0]. The catalysts contained a Group I-III compound and a Group IVA-VI or VIII transition metal compound

L5 ANSWER 29 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1975:90646 CAPIUS
DOCUMENT NUMBER: 82:90646

TITLE: Catalyst for gaseous production unsaturated **esters** containing titanium (oxide), palladium, alkali metal carboxylate and a **carrier**
INVENTOR(S): Nakamura, Seishiro; Kushida, Koichi; Yasui, Akio
PARENT ASSIGNEE(S): Kuraray Co., Ltd.
SOURCE: Jpn. Tokkyo Koho, 3 pp. Division of Japan. 72 45,324 (CA 78: 57793a).
CODEN: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. JP 49030809
 PRIORITY APPL. INFO.:
 AB A catalyst for **gas-phase ester** manufacture contains 0.1-10 wt% metal or Pd metal, 0.1-20 alkali metal carboxylate, and 0.01-50 weight % Ti metal or oxide, with optional Au or Ag additives, on an active C, Al₂O₃, SiO₂, or aluminosilicate **carrier**. The Ti is added to the known Pd catalyst by conventional methods, giving greater tolerance to severe reaction conditions. The **esters**, e.g., vinyl acetate, are prepared by reaction of alkene with **carboxylic acid** and oxygen at 80-200° under 1-10 atmospheric pressure.

L5 ANSWER 30 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1974:412587 CAPLUS
 DOCUMENT NUMBER: 81:12587
 TITLE: Organic **esters**
 INVENTOR(S): Nakamura, Seishiro; Yasur, Teruo
 PATENT ASSIGNEE(S): Kuraray Co., Ltd.
 SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.
 CODEN: JKKXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO. JP 48103509
 JP 56052008
 PRIORITY APPL. INFO.:
 AB **Olefins**, **esters** of unsatd. alcs., or alkylbenzenes were treated with O and **carboxylic acids** to give organic **esters** in the presence of a Pd catalyst on a **carrier**, in which the volume of pores having a diameter ≥1000 Å occupied 280% of the total volume of pores. Thus, a **gas** mixture containing butadiene, O, AcOH, and N (30:10:20:40) was passed at 140° and 5 l./hr over a Pd catalyst on an alumina **carrier**, in which the volume of pores with a diameter ≥1000 Å occupied 98% of the total pore volume, to give 1,4-diacetoxy-2-butene (I), 3,4-diacetoxy-1-butene (II), butadienylacetate (III), resp., at 52, 2, and 3 g/catalyst 1-hr after 1 hr reaction. Similarly, with a silica **carrier** methacryl acetate and 2-methylene-1,3-diacetoxy-propane were formed from a mixture of isobutylene, O, AcOH, and N. Also, 1-butene, 2-butene, AcOH, and air gave I, II, 3-acetoxy-1-butene, 1-acetoxy-2-butene, and III. Similarly, 1-acetoxy-2-butene, AcOH, O, and N gave I, II, and III.

L5 ANSWER 31 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1974:127889 CAPLUS
 DOCUMENT NUMBER: 80:127889
 TITLE: Identification of hydroxy(acetoxy)alkanes by **gas-liquid chromatography** combined with ir and NMR spectroscopy
 AUTHOR(S): Nikitina, N. S.; Mysak, A. E.; Veretenova, T. N.; Tikhonov, V. P.; Lebedev, E. V.
 CORPORATE SOURCE: Vses. Nauchno-Issled. Proektno-Konstr. Inst. Neftpererab. Neftekhim. Prom., Kiev, USSR
 SOURCE: Neftpererabotka i Neftekhimiya (Kiev) (1973), No. 9, 88-91
 CODEN: NEENBY; ISSN: 0548-1406
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian
 AB During the preparation of C8-18 1,2-alkanediols (I) from α-

olefins by the Prileshaev reaction, the 1-acetoxy-2-hydroxyalkane (II) and 1-hydroxy-2-acetoxyalkane (III) intermediates and I were separated by **gas chromatog.** on a 5% SE-30/Chromosorb W column by using He **carrier gas** and a flame ionization detector. For all compds., the retention time increased with increasing C number. On the basis of ir and NMR spectra, the order of elution was I, III, and II off each group with a given C number

L5 ANSWER 32 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1974:15437 CAPLUS
 DOCUMENT NUMBER: 80:15437
 TITLE: Unsaturated **esters**
 INVENTOR(S): Saito, Toshihiro; Takano, Misao; Moriyama, Shigeru; Murayama, Hiroshi
 PATENT ASSIGNEE(S): Tekkoshu Co., Ltd.
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
 CODEN: JKKXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO. JP 48049711
 JP 52029294
 PRIORITY APPL. INFO.:
 AB Vapor phase reaction of a lower unsatd. hydrocarbon with an acid in the presence of O over a palladium [7440-05-3] and gold [7440-57-5] catalyst optionally containing uranium [7440-61-1] and **supported on** a zinc spinel, gave unsatd. **esters**. Thus, Al₂O₃ was saturated with aqueous Zn(NO₃)₂ solution and heated to form a spinel which was soaked with HCl containing FeCl₂, AuCl₃, and UOAc₃. The treated spinel was treated further with alkaline NH₄H₂O and aqueous AcOK to give a catalyst containing Pd 1, Au 0.1, U 0.1, and AcOK 4.7 weight%. A mixture of propylene [115-07-1] 0.914, O 0.441, and **acetic acid** [64-19-7] 0.513 mole/hr was passed over the catalyst at 160 deg. to give allyl acetate [591-87-7]. Vinyl acetate [108-05-4], vinyl propionate [105-38-4], and allyl propionate [2408-20-0] were similarly prepared

L5 ANSWER 33 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1972:526021 CAPLUS
 DOCUMENT NUMBER: 77:126021
 TITLE: Silica **supports** for palladium catalysts for **olefin** oxyacylation
 INVENTOR(S): Fernholz, Hans; Wunder, Friedrich; Schmidt, Hans Joachim
 PATENT ASSIGNEE(S): Farbwerke Hoechst A.-G.
 SOURCE: Ger. Offen., 12 pp.
 CODEN: GWXXBX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO. DE 2100778
 DE 2100778
 NL 7200013
 ES 398552
 ZA 7200037
 AU 7237544
 PRIORITY APPL. INFO.:
 AB **Olefins** were reacted with O and **carboxylic acids** to give organic **esters** in the presence of a Pd catalyst on a **carrier**, in which the volume of pores having a diameter ≥1000 Å occupied 280% of the total volume of pores. Thus, a **gas** mixture containing butadiene, O, AcOH, and N (30:10:20:40) was passed at 140° and 5 l./hr over a Pd catalyst on an alumina **carrier**, in which the volume of pores with a diameter ≥1000 Å occupied 98% of the total pore volume, to give 1,4-diacetoxy-2-butene (I), 3,4-diacetoxy-1-butene (II), butadienylacetate (III), resp., at 52, 2, and 3 g/catalyst 1-hr after 1 hr reaction. Similarly, with a silica **carrier** methacryl acetate and 2-methylene-1,3-diacetoxy-propane were formed from a mixture of isobutylene, O, AcOH, and N. Also, 1-butene, 2-butene, AcOH, and air gave I, II, 3-acetoxy-1-butene, 1-acetoxy-2-butene, and III. Similarly, 1-acetoxy-2-butene, AcOH, O, and N gave I, II, and III.

PRIORITY APPL. INFO.: DE 1969-1808610 A 19681113
AB Vinyl acetate (I), propionate, or isobutyrate and alkyl acetate are prepared from **olefins**, C2-20 aliphatic and aromatic **carboxylic acids**, and mol. O with a metal ion catalyst and catalyst activators.

15 ANSWER 36 OF 52
 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1969-513096 CAPLUS
 DOCUMENT NUMBER: 71:113096
 TITLE:
 Mucic acidic constituents of vetiver oil. II.
 Cyclooctaamphenic and pivalocyclooctaamphenic acids
 Kida, Fusao, Sakuma, Raiko, Uda, Hiashiki, Yoshikoshi,
 Akira,
 AUTHOR(S):
 CORPORATE SOURCE: Tohoku Univ., Sendai, Japan
 SOURCE: Tereshedron Letters. (1969). (37), 3169-72
 CODEN: TELLFV; ISSN: 0040-4039

and submitted to mild alkaline hydrolysis to yield the major acid, cyclohexanecarboxylic acid, C₁₅H₂₂O₂ (I, R = CO₂H) (II), m. 151.5-2.5°, [α]_D²⁰ -14.7° (c 1.25, CHCl₃), and the minor acid epicyclo-copacampheic acid (I, R = CO₂H) (III), m. 168.0-8.5°, [α]_D²⁰ 5D 78.3° (c 3.6, CHCl₃).

Spectral and chemical evidence indicated that I are saturated tetracyclic compds.

with one secondary and 2 tertiary Me groups with a probable tricyclic carbon framework, both derived from the same parent hydrocarbon I (R = Me) (IV). The mixed acids treated with Pb(OAc)₄-LiCl followed by dehydrochlorination of the epimeric Cl derivs. (I, R = Cl) gave a mixture of geometrically isomeric olefins (V). The N.M.R. chemical shift of the values for the secondary Me group in the spectra of the Cl derivs. and V showed the existence of the >CHMeCO₂H group in II and III. V hydroxylated and submitted to glycol cleavage gave a single cyclic ketone (VI), v 1701, 1420 cm.⁻¹. The Baeyer-Villiger oxidation of VI afforded an α-lactone, C₁₂H₁₆O₂, m. 109-11°, subsequently transformed into an oxo carboxylic acid (VII), C₁₂H₁₆O₃, m.

114.0-15.5°, by saponification followed by Jones oxidation. The spectral properties of VII and its Me ester verified the tricyclicanone structure and it was presumed that the C skeleton of I and II, including the 3-carbon side chain would be that of cyclosativene (VIII, R = iso-Pr, R₁ = H) (IX). According to the reaction sequence: -CO₂Me → -CH₂OH → p-MeC₆H₄SO₃CH₂- → -Me, II and III gave the same hydrocarbon IV whose IR and N.M.R. spectra indicated stereoisomeric with IX with respect to the iso-Pr group, a supposition confirmed by conversion of I and III to IX by decarboxylative acetoxylation to I (R = OAc) with Pb(OAc)₄; hydrolysis with alc. KOH followed by Jones oxidation to VIII (R = H, R₁ = Ac); epimerization with NaOMe to yield the isomer VIII (R = Ac, R₁ = H); and Wittig reaction with Ph₃P:CH₂ to VIII (R = MeC=CH, R₁ = H), followed by catalytic hydrogenation over (Ph₃P)3RhCl as catalyst. The resulting hydrocarbon C₁₅H₂₄, [α]_D²⁰ 67.8° (c 1.15, CHCl₃), m.p. 204 was spectroscopically identical with (-)-cyclosativene (IX), [α]_D²⁰ 94.1°, leading to the structures of II and III. The strongly pos. sign of the Cotton effect in the O.R.D. of VI supported the absolute stereochemistry of the C skeleton of VI.

L5 ANSWER 37 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1969:402987 CAPLUS

DOCUMENT NUMBER: 71:2987

TITLE: Esters of unsaturated alcohols

INVENTOR(S): Capp, Clifford W.; Durston, Peter J.

PATENT ASSIGNEE(S): BP Chemicals (U.K.) Ltd.

SOURCE: Brit., 2 pp.

CODEN: BRXXAA

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1148583	---	---	---	---
AB	Vinyl acetate (I) is manufactured by combining C ₂ H ₄ , O, and AcOH in the vapor phase at elevated temps. in the presence of Pd deposited on Al ₂ O ₃ , Fe oxide, or Al Mg spinel supports. The exhausted catalysts are reactivated by treatment in 2N NaOH. Thus, 8-16-mesh Al ₂ O ₃ was treated with 1:1 PdCl ₂ -HCl solution, dried at 140°, and reduced with alkaline N ₂ H ₄ to yield 2% Pd/al ₂ O ₃ catalyst (II). C ₂ H ₄ 0.29, O 0.06, and AcOH 0.14 mole/hr. were combined at 140° in the presence of II to yield 0.21 mole/l./hr. I after 4 hrs. and 0.13 mole/l./hr. for the next 190 hrs., before falling to 0.02 mole/l./hr. after 217 hrs. The spent II was treated with 3 successive ams. of 2N NaOH, followed by 6 successive washings with cold H ₂ O, and drying at 140° to give a reactivated-II that yielded 0.18 mole/l./hr. I for the next 236 hrs. Uses of other	19690416	GB 1966-28591	19660625

Pt-group metals on aluminosilicates, heavy metal oxides, metal oxide-Al₂O₃ complexes, carbon, or pumice, and catalyst reactivation with aqueous solns. of KOH, Na₂CO₃, or K₂CO₃ and alc. solns. of Na-(or K) methoxide or methoxide, were claimed.

L5 ANSWER 38 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1969:116669 CAPLUS

DOCUMENT NUMBER: 70:116669

TITLE: Finely divided metals of the platinum group

PATENT ASSIGNEE(S): Monsanto Co.

SOURCE: Brit., 7 pp.

CODEN: BRXXAA

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1146530	---	---	---	---
DE 1533102	---	19690326	GB 1966-22105	19660518
PRIORITY APPLN. INFO.: DE	---	---	---	---
AB	A method of producing finely divided metals of the Pt group, the products obtained, supported catalysts made therefrom and their use for hydrogenation and dehydrogenation are described. The finely divided metal is obtained by reducing a solution of a salt of the metal in the presence of an alc., acetal, ketone, ether, carboxylic acid ester, or olefin, which has a reducing action on the salt and forms a soluble complex with the metal, by the action of H ₂ on the solution in the absence of a borohydride, or by thermally decomposing the complex, thereby producing the metal in a zero-valent state. The process may be carried out in the presence of a non-reducing solvent for the metal salt. Particularly suitable reducing agents (solvents) for this process are the cyclic ethers, especially propylene oxide. In carrying out the treatment with H ₂ , the solution may be preheated. The thermal decomposition of the complex	CH	19650518	---

may be effected before or after removal of excess reducing agent and may occur slowly at room temperature; usually it is sufficient to heat the solution for 120 min. up to 90° to obtain a quant. suspension of the Pt. metal. The finely divided Pt metals can be deposited on supports, especially Al₂O₃ which have been subjected to phys. or chemical pretreatment such as glowing, action of acids, or with superheated H₂O-vapor. The metals are established on the support by steeping or by pouring the solution onto the support. Solvent is then removed in vacuo at room temperature with effective stirring. The metal compound which remains on the support contains enough, probably complex-linked, reducing agent to give the zero-valent metal on heating up to higher temps. (e.g., to 150-250° for hydrogenation catalysts) preferably under reduced pressure. For example, to prepare finely divided Pt on a support, the pulverized or granulated support is heated in a vacuum (0.004 mm.) at 150° for 2 hrs. The Pt salt is subject to the same treatment. A solution containing (1-2) + 10-2 mole Pt salt/mole solution is prepared in an atmosphere of Ar by using the cited reducing agent. The solution is slowly added, with cooling, either to the cooled powdery support or to the granulated support previously freed of air by evacuating until the carrier mass is well saturated with it. The excess solvent is distilled immediately with stirring in a vacuum at 25° and the catalyst is heated in the vacuum at the indicated after-treatment temperature for 1 hr. After cooling the catalyst to room temperature, the catalyst is used approx. 24-48 hrs. later. The air is admitted. The catalysts prepared with different reducing agents and activities of such catalysts prepared with different reducing agents and

supports are tabulated.

L5 ANSWER 39 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1969:105986 CAPLUS
DOCUMENT NUMBER: 70:105986

TITLE: Carboxylic acids and their unsaturated

esters

INVENTOR(S): Sennewald, Kurt; Vogt, Wilhelm; Erpenbach, Heinz;

Glaser, Hermann

Knapsack A.-G.

S. African, 18 pp.

CODEN: SEFXAB

PATENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ZA 6802010	----	-----	-----	-----
DE 1618591	----	19680829	-----	-----
FR 1567312	----	-----	-----	-----
GB 1189091	----	-----	-----	-----
RO 52442	----	-----	-----	-----
US 3631079	----	19711228	-----	-----
US 19680322	----	-----	-----	-----
US 19670405	----	-----	-----	-----

PRIORITY APPLN. INFO.:
AB An aldehyde is partially or completely substituted for its corresponding acid in the gas-phase reaction of an olefin, the carboxylic acid, and O in contact with a Pd-containing catalyst at elevated temperature to give an unsatd. ester of the acid. The aldehyde is simultaneously oxidized in the process, thus reducing the cost of oxidation of the aldehyde to the acid in an addnl. step. Thus, the catalyst was prepared by impregnating 1 kg. of a ball-shaped silicic carrier with an aqueous solution of 8 g. PdCl₂ and 3 g. H₂NC₁₄, drying the mixture with agitation, pouring into a 4-5% H₂NNH₂.H₂O solution at 40°, and, after reduction was complete, pouring off the supernatant liquid, washing the catalyst with distilled H₂O, impregnating with 1% aqueous KOAc

solution, decanting the solution, and drying the catalyst under reduced pressure at 60°. The catalyst (1 l.) was placed in a steel tube, and 500 l./hr. C₂H₄, 500 g./hr. HOAc, and 25 g./hr. AcH was passed over the catalyst at 6 atmospheric absolute. The reaction gas was cooled to 0° to give a mixture comprising 70% (428 g.) HOAc, 23.4% vinyl acetate, 6.45% H₂O, and approx. 0.2% AcH. Approx. 32.1 g. fresh HOAc was formed by AcH oxidation, and approx. 30.9% of the HOAc obtained by oxidation had reacted. Vinyl isobutyrate was similarly prepared

L5 ANSWER 40 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1969:96179 CAPLUS
DOCUMENT NUMBER: 70:96179

TITLE: Olefins

PATENT ASSIGNEE(S): Stamlicarbon N. V.

SOURCE: Meth. Appl., 7 pp. Addn. to Neth. Appl. 66 08559

CODEN: NAXXAN

PATENT TYPE: Patent

LANGUAGE: Dutch

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
NL 6706640	----	-----	-----	-----
DE 1643077	----	19681113	-----	-----

GB 1154517

AB Catalysts useful for preparing unsatd. esters from saturated organic acids, olefins, and mol. O are prepared by impregnating a carrier with a solution of a Pd salt and Pt, or Rh salt, and then drying, hydrolyzing, and reducing the impregnated carrier mass.

Thus, catalysts are prepared by impregnating Al₂O₃ with solns. of Pd salts and Pt salts. The impregnated mass is then dried, hydrolyzed by treatment with boiling NaHCO₃ solution, and reduced. These catalysts are used in the synthesis of vinyl acetate (I) by passing 120 l./hr./l. of catalyst of a 1:1.0.4 molar C₂H₄-HOAc-O gas mixture over the catalyst at 1 atmospheric For example a catalyst containing 2.9% Pd and 29.8 and 91%, resp., in 18 days. A catalyst containing 2.0% Pd and no Pt gave 15.1% conversion and 92% yield in 1 day and 7 and 90%, resp., in 4 days at 118°. A catalyst containing no Pd and 3.0% Pt gave resp. values of 10.5 and 9% in 1 day at 111°. Best results (42.5 and 88%, resp.) were obtained with 2.2% Pd and 0.03% Pt in 1 day at 120°. A catalyst containing Rh was also used. These catalysts have excellent retention of activity over long operating times, and give good conversions to the desired product.

L5 ANSWER 41 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1969:96171 CAPLUS
DOCUMENT NUMBER: 70:96171

TITLE: Gas-phase synthesis of vinyl esters

from ethylene, oxygen, and carboxylic acids

with a palladium-alkali metal acetate catalyst

Brit., 10 pp.

CODEN: BRXXAA

PATENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.

KIND

DATE

APPLICATION NO.

DATE

GB 1142250

DE 1568939

FR 1520433

US 3567767

19710302

US

JP

19661209

19651217

PRIORITY APPLN. INFO.:
AB Vinyl esters were prepared by continuous gas phase synthesis from C₂H₄, O, and an organic carboxylic acid, using a catalyst system having an improved and high catalytic activity and which minimizes the rate of loss of its catalytic activity by performing the reaction under a low partial pressure of O and organic carboxylic acid. Thus, bead alumina having 99.3% Al₂O₃, a 210 m.²/g. surface area, and 10-14 mesh were calcined 3-5 hrs. in air at 550-1300°. The bead alumina was prepared by adding 175 g. CaCO₃ wetted with 30 cc. water to a solution of 342 g. Al₂(SO₄)₃ dissolved in 800 cc. water. CO₂ was generated and CaSO₄ was precipitated. CaSO₄ was filtered to give Al₂O₃.l.lSO₃ (I). The liquid had 3.38 pH. I was dropped through an 85° heated glass tube to give a gel, which was purified and calcined 5 hrs. at 500°. The calcined carrier (35 parts) was added to 50 parts water containing 0.3 part HCl and 1.3 part PdCl₂ and the system dried. PdCl₂ was reduced by dropping a reducing liquid on the dried catalyst system. The liquid was prepared by adding 2.5 parts of 80% hydrazine hydrate solution to 50 parts N NaOH solution. The reduced catalyst was water-washed and placed in 10 parts water containing 0.7 parts AcOH and dried. The catalyst (35 g.) was placed in a glass tube heated to 110° and a gaseous mixture containing C₂H₄, O, AcOH (4:1:2 molar ratio) was passed over the catalyst at 8 l./hr. In an experiment, where the calcination temperature for the carrier was 900° and the surface area of the carrier was 120 m.²/g., the production rate of vinyl acetate (II) was 40.1 g. II/l./catalyst hr., the II selectivity, 94%, and AcH selectivity, <0.1.

L5 ANSWER 42 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1966:27122 CAPLUS
DOCUMENT NUMBER: 64:27122
ORIGINAL REFERENCE NO.: 64:4949d-e
TITLE: Ethylenically unsaturated **esters**
PATENT ASSIGNEE(S): Pullman Inc.
SOURCE: 5 pp. Patent
DOCUMENT TYPE: Unavailable
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1007815	---	---	19651022	---

PRIORITY APPLN. INFO.: 19601223
AB Olefins halogenated on one of the doubly bound C atoms react in liquid or vapor phase, under anhydrous conditions, with **carboxylic acids** in the presence of halides of Group VIII metals to form unsatd. **esters**. The halides may be used in combination with a **support carrier**, or diluent, such as alumina, silica gel, and alundum. The greater the ratio of the catalyst to olefin reactant the faster the rate of reaction. Reaction takes place between 70 and 300°F. and mostly at the atmospheric pressure. Thus, 3.56 g. PdCl₂ and 11.36 g. Na₂HPO₄ is kept for 2-4 days in vacuo at 375°F., cooled to room temperature, 50 cc. of isooctane added, and the vessel flushed with vinyl chloride at 720 mm., with shaking. The reaction vessel is charged with 3.76 g. AcOH and shaken at room temperature for 4 hrs. The yield of vinyl acetate is 2.6 mole-% on the basis of PdCl₂.

L5 ANSWER 43 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1966:26837 CAPLUS
DOCUMENT NUMBER: 64:26837
ORIGINAL REFERENCE NO.: 64:4887g-h, 4888a-h, 4889a

TITLE: Solvolytic rearrangement of the 2-(1-cyclopentenyl)ethyl system
AUTHOR(S): Closson, W. D.; Kwiatkowski, G. T.
CORPORATE SOURCE: Columbia Univ.
SOURCE: Tetrahedron (1965), 21(10), 2779-89
CODEN: TETRAH; ISSN: 0040-4020

DOCUMENT TYPE: Journal
LANGUAGE: English
GI For diagram(s), see printed CA Issue.
AB Cf. CA 62, 6357d. The acetoysis of 2-(1-cyclopentenyl)ethyl bromobenzene-sulfonate (I) was investigated. Reduction of 1-cyclopentenylacetic acid in tetrahydrofuran with LiAlH₄ yielded 90% 2-(1-cyclopentenyl)ethanol (II), b₃ 65-6°, n_D 20 1.4781. Similar reduction of cyclopentylacetic acid gave 90% yield of 2-cyclopentylethanol (III), b₃ 77-8°. Conversion of 17 g. II gave 10 g. of the corresponding bromide, b₀ 5.43-4°. The bromide (57 millimoles) and 70 millimoles KCN in 25 ml. HOCH₂CH₂OH heated 2.5 hrs. at 100° and the cooled solution poured into saturated aqueous NaCl, extracted with Et₂O, and the residue on evaporation heated 20 hrs. at 95° in 200 ml. 20% aqueous KOH, the cooled solution washed with Et₂O and acidified with aqueous HCl, extracted with Et₂O, and the isolated 3-(1-cyclopentenyl) **propionic acid**, (2.5 g., m. 58-61°), reduced with LiAlH₄ in tetrahydrofuran yielded 84% 3-(1-cyclopentenyl)-propanol (IV), b₃ 66-7°. Preparation according to Alibisetti, et al. (CA 51, 1041d) gave 3-methylenecyclohexanol (V), b₄ 97.5-8.0°. Zn-Cu couple (2 g.) in 30 ml. dry Et₂O and 11.6 millimole 2-methylenecyclopentanone in 7 g. CH₂I₂ stirred 16 hrs. and diluted with 5 ml. saturated aqueous NH₄Cl, the organic layer and Et₂O washings washed, dried, and concentrated and the residual oil kept 16 hrs. in 10 ml. saturated MeONa

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under N, poured into saturated aqueous NaOH, and extracted with Et₂O yielded spiro[2.4]heptan-4-ol (VI), b₂ 75-8°. Similar conversion of 1-cyclopentenylmethanol gave 45% yield of bicyclo[3.1.0]hexane-1-methanol (VII), b₄ 96-8°; p-nitrobenzoate (VIII) m. 44-5°. Cyclopropylcarbinol and p-O₂NC₆H₄COCl in C₅H₅N gave cyclopropylcarbinyl p-nitro-benzoate, m. 56-7°. Spiro[2.4]heptan-4-yl p-nitrobenzoate (IX) was prepared by Appelquist and Landgrebe (CA 60, 1310d). Treatment of the appropriate alc. with the requisite sulfonyl chloride gave I (63%), 2-cyclopentylethyl p-bromobenzenesulfonate (X, oil, 62%), 3-(1-cyclopentenyl)propyl p-bromobenzenesulfonate (XI, m. 40.0-1.5°, 79%), and 3-methylenecyclohexyl tolylsulfonate (XII, oil, 75%). I (33.2 millimoles) and 40 millimoles NaOAc in 500 ml. AcOH heated 2 hrs. at 100° and added to 1 l. H₂O, extracted continuously with C₅H₁₂ and the extract slowly distilled to 250 ml. concentrate, submitted

to analysis on an 8-ft. 1,2,3-tris-(2-cyanoethoxy)propane column at 125° to show the presence of 10% of a mixture of 2 **olefins** (major constituent tentatively identified as 3-methylenecyclohexene, the remaining C₅H₁₂ evaporated and the acetate mixture (87.4% yield) reduced with LiAlH₄ in Et₂O gave a mixture of alcs., analyzed by **gas chromatography** to show the presence of VI 11.2, bicyclo[3.2.0]heptan-1-ol (XII) 21, V 56.9, VII 6.2, and II 4.7%. XIII was identified by m.p. (42-3°) and ir and N.M.R. spectra, and showed a major peak at m/e 84, apparently due to loss of H₂C:CH₂. XII (4.14 millimoles) and 11 ml. 0.45M NaOAc-AcOH in 100 ml. AcOH heated 3 hrs. at 100° yielded 37% **olefins** (4:1 ratio), minor component probably 3-methylenecyclohexene) and 63% 3-methylenecyclohexyl acetate (XIV). The rate of acetoysis at 80° was only 5.07 ± 0.05 + 10-5 sec.-1, about half that of cyclohexyl tolylsulfonate (10.7 + 10-5 sec.-1) under the same conditions. IX (0.23 millimole) and 0.40 millimole 2,6-lutidine in 25 ml. 60% aqueous Me₂CO heated 4 hrs. at 100° in a glass ampul, the Me₂CO evaporated, and the aqueous solution extracted with Et₂O, washed

(cold dilute HCl, aqueous Na₂CO₃), and the dried extract concentrated to 4 ml., examined by **gas chromatography** to show the presence of 60.6% VI, 25.4% XIII, and 14% V, and evaporated gave an oil; ir spectrum contd. bands present in the ir spectra of each of the 3 alcs. XIII (4.5 millimoles) and 1 millimole p-O₂NC₆H₄CO₂H in 100 ml. 60% aqueous Me₂CO heated 2.5 hrs. at 100° in a glass bomb gave a mixture containing 63.5:36.5 XIII-V. Solvolysis of 5.37 millimoles VIII with 8.72 millimoles 2,6-lutidine in 100 ml. 60% aqueous Me₂CO 60 hrs. at 100° gave 2.5% **olefin**, 1.5% VI, 4.3% XIII, 82.8% V, and 8.9% VII. The rates of solvolysis of p-nitrobenzoate **esters** of IX, VIII, and related p-nitrobenzoate **esters** were tabulated. Acetoyses and ethanolyses were carried out by the ampul technique. All kinetic solns. were 0.036M in NaOAc and 0.030M in sulfonate **ester**. The aliquots were acidified with standard HClO₄-MeOH and back-titrated with 0.020N NaOAc-AcOH (acetolyses) or 0.015M NaOMe-MeOH (ethanolyses), using bromothymol blue indicator. Rates of solvolyses of I, X, 2-(2-cyclopentenyl)ethyl bromobenzenesulfonate (XV), 3-cyclopentylpropyl bromobenzenesulfonate (XVI), and XI were tabulated. The ethanolysis rate of I was essentially that of 2-substituted ethyl brosylates X and XV. On changing to AcOH, the reactivity of I increased slightly, whereas that of X and XV decreased sharply. The insensitivity of I to change in nucleophilicity of solvents is fairly typical of systems that solvolyze with participation of double bonds. The lack of any significant difference in acetoysis rates between XI and XVI supported the conclusion that inductive effects were minor and that anchimeric acceleration in the acetoysis of I is only about 40-fold. Comparison with other primary homoallylic systems indicated that a better value for anchimeric acceleration in the 2-cyclopentenylcarbinyl system is 50-60 fold. The enhanced rate and production of rearranged products on acetoysis of I clearly indicated participation of the double bond but direct ionization to an intermediate cation capable of yielding all of the

rearranged acetates seemed unlikely. The first-formed intermediate should account for the rate enhancement and the bicyclobutonium ion (XVII) would do so and would also explain formation of spiro[2.4]-heptan-4-yl acetate (XVII) and bicyclo[3.2.0]heptan-1-yl acetate (XIX). The unrearranged 2-(1-cyclopentenyl)ethyl acetate (XX) probably came from a competing solvolytic displacement. The other two rearranged acetates, bicyclo[3.1.0]-hexane-1-methyl acetate (XXI) and XIV cannot be obtained from XVII and a bridged homallylic ion (XXII) or a bridge cation (XXIII) were postulated as precursors with conversion to ion (XXIV) involving only minor changes in bond lengths and conformation. Accordingly the acetalysis of I was rationalized schematically as: I \rightarrow XVII + .dihaw. XXII (or XXIII) \rightarrow XXIV \rightarrow XIV, XVII \rightarrow XVIII + XIX, XXII (or XXIII) \rightarrow XXI, XXII (or XXIII) \rightarrow XIV. Other cations could also be present but the proposed scheme is sufficient to account for the products and to provide a reasonable explanation for the reactivities of the related systems. This general reaction class may have synthetic utility. The biol. extensive 2-(β -indolyl)ethyl system is analogous in many ways to the 2-(1-cyclopentenyl)ethyl structure.

L5 ANSWER 44 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1966:11686 CAPLUS

DOCUMENT NUMBER: 64:11686

ORIGINAL REFERENCE NO.: 64:2149e-h,2150c

TITLE:

Extension of sugar chains through acetylenic intermediates

Horton, D.; Hughes, J. B.; Tronchet, J. M. J.

Ohio State Univ., Columbus

Chemical Communications (London) (1965), (20), 481-3

CODEN: CCMA8; ISSN: 0009-241X

DOCUMENT TYPE:

Journal

Language:

English

CASREACT 64:11686

GI For diagram(s), see printed CA Issue

AB Oxidation of 1,2,3,4-di-O-isopropylidene- α -D-galactopyranose with MeSO₃N'-dicyclohexylcarbodiimide, followed by ethynylation of the resultant aldehyde gave the 6-epimeric mixture (I). The preponderant epimer (m. 130-1°) was reduced to the corresponding olefin, m. 106-08°. Similarly, ethynylation of periodate-oxidized 1,2-O-isopropylidene- α -D-glucopyranose gave a mixture of 5-epimers, the D-glucose-epimer gave a crystalline 3,5-dibenzoate, m. 191-3°. Reduction of the latter to the olefin, m. 143-5°, followed by ozonolysis, reduction, and hydrolysis gave glucose. The 3-epimeric heptynes (II) formed by ethynylation of 2,3,4,5-di-O-isopropylidene-aldehyde-L-arabinose were separated by gas-liquid chromatography (9.1.c.) to give the D-glucose-isomer, m. 67-9°, [α]22D 6.5° (CHCl₃) and the D-manno-isomer, [α]22D -24° (CHCl₃), in 3:2 proportion: the structures were established by degradation. The 3-acetate of II was treated with bis(1,2-dimethylpropyl)borane (IV) in diglyme, followed by H₂O₂, to give the 2,3-trans-unsaturated aldehyde (III), b.p. 65°. Similarly, ethylation of 1,2-O-isopropylidene-D-glycerinaldehyde gave a 3:2 mixture of 3-epimeric pentyne derivs., separable as the 3-acetates (V) by g.l.c. One epimer had [α]20D -41° (CHCl₃), and the other [α]30D 50° (CHCl₃). Treatment of V with IV, followed by H₂O₂, gave trans-2,3-dideoxy-4,5-O-isopropylidene-aldehyde-D-glycero-2-pentose, the 5-carbon analog of III. Structures given were supported by ir and N.M.R. data.

L5 ANSWER 45 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1964:484851 CAPLUS

DOCUMENT NUMBER: 61:84851

ORIGINAL REFERENCE NO.: 61:14839c-g

TITLE:

Modification of textile fiber properties by

radiation-induced graft copolymerization

Armstrong, Arthur A., Jr.; Walsh, William K.;

Rutherford, Henry A.

AUTHOR(S):

SOURCE:

DOCUMENT TYPE:

Journal

Language:

Unavailable

AB The modification of fiber properties by exposure to radiation and by graft copolymerization (GP) is described. The radiation source was a 60Co γ -cell 220. The predominant effect of radiation on fibers was degradation. No improvement of the phys. properties of textile fibers by radiation alone was indicated. Vapor-phase GP to textile fibers initiated by γ radiation was investigated for (a) variables affecting the rate of GP; (b) modification of fiber properties by GP; and (c) kinetics and diffusion in the vapor-phase GP process. The effects of process variables on the rate of grafting were: (1) higher carrier-gas flow rates increased the rate of grafting; (2) higher temps. decreased the rate of grafting; (3) the presence of a 3rd component (e.g. H₂O, MeOH, or AcOH) increased the rate of grafting to acetate and nylon, and was necessary for grafting to cotton, rayon, and wool; (4) O inhibited polymerization; (5) the rate of grafting depended on the fiber and the monomer; the order of reactivity for fibers was acetate > polypropylene > nylon > cotton > rayon > wool > polyester > acrylic; the order of reactivity of the monomers studied was Me methacrylate > Et acrylate > acrylonitrile > butadiene > vinyl acetate > Me acrylate > vinyl chloride > styrene; (6) chemical modification of the cellulose increased the rate of grafting to cotton; (7) the lifetimes of free radicals produced by irradiation were long enough to use post-irradiation techniques. Radiation-induced vapor-phase GP of vinyl monomers to textile fibers was studied as a means to improve fiber properties with the following results: (1) cotton was completely resistant to microorganism attack and had increased resistance to heat after grafting with acrylonitrile; (2) polypropylene fiber was dyeable with disperse dyes after grafting with vinyl acetate, Me acrylate, and Et acrylate; (3) nylon and polypropylene had improved weather resistance after grafting with styrene; (4) cotton cloth had improved crease recovery after grafting with allyl acrylate, followed by cross-linking by radiation and grafting with methylacrylamide; (5) nylon had increased dimensional stability at 290° after grafting with allyl acrylate, acrylic acid, or a combination of acrylic acid with a cross-linking monomer, and had 30-50% increased modulus after grafting with acrylic acid alone or in combination with a cross-linking monomer. A preliminary study of the effects of diffusion and kinetics on the vapor-phase GP of acrylonitrile to various textile fibers showed that the effects are complex and cannot be represented by simple math. solutions. Empirical relations were obtained which adequately represented the more complex cases.

L5 ANSWER 46 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1964:24794 CAPLUS

DOCUMENT NUMBER: 60:24794

ORIGINAL REFERENCE NO.: 60:4439b-d

TITLE:

Reaction gas chromatography

Drawert, Friedrich

Abhandl. Deut. Akad. Wiss. Berlin, Kl. Chem., Geol.

SOURCE:

Biol. (1962), (1), 9-18

DOCUMENT TYPE:

Journal

Language:

Unavailable

AB A reactor is interposed between the carrier gas supply and the anal. column in which the sample is transformed chemical to yield better separation of alcs. and polyols in aqueous solution if the reaction temperature

permits, the reactor tube can be placed in the oven compartment in series with the anal. column. A sep. thermostat is used for high-temperature reactions. H₂O can be removed by a CaH₂ column ahead of the anal. column. Aqueous solns. of lower alcs. are acidified with either tartaric or oxalic acid and passed through a column containing a mixture of NaNO₂ and support material, or a NaNO₂-containing solution of the alcs. is passed through a reactor tube containing one of the acids. The nitrous acid

esters are separated much more readily than the alcs. When alcs., their mixture, or aqueous solns. are sprayed on hot acidic surfaces, dehydration yields the corresponding **olefins**, which can be readily separated. The method was used for blood alc. detns. Some alcs. can be determined by first hydrogenating over Raney Ni at 170-200° to the hydrocarbon. Bis(alkyl xanthogenato)nickel(II) complexes are split with dimethylglyoxime, and the alcs. liberated by acidification are determined by the **olefin** method. 2-Alkoxy-2-mercaptobenzothiazolines are readily converted to 2-mercaptobenzothiazolines by splitting off an alc. which is determined as above. Aqueous glycerol is converted by HI to iso-PI, which is determined as propane by the hydrogenation method.

L5 ANSWER 47 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1963:29840 CAPLUS
DOCUMENT NUMBER: 58:29840
ORIGINAL REFERENCE NO.: 58:5018d-e
TITLE: Gas-liquid chromatography of synthetic carboxylic acids and the corresponding alcohols
AUTHOR(S): Vasilescu, V.
CORPORATE SOURCE: Inst. Verfahrntechnik Org. Chem., Leipzig, Germany
SOURCE: Abhandl. Deut. Akad. Wiss. Berlin, Kl. Chem., Geol., Biol. (1959), (No. 9), 136-53
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB The products of paraffin oxides., namely C4-12 **carboxylic acids** and C8-14 alcs. were separated by using high vacuum grease Number 20 (Carl Zeiss, Jena) on ground Kaolin "MEKA" as a **carrier**. The **carboxylic acids** were converted to the **Me esters** with CH2N2 and the alcs. were preferably acetylated before analysis.

L5 ANSWER 48 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1961:101313 CAPLUS
DOCUMENT NUMBER: 55:101313
ORIGINAL REFERENCE NO.: 55:19070d-f
TITLE: Catalysts
INVENTOR(S): Dowden, Dennis Albert; Caldwell, Alexander M. U.
PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 977324	---	19610328	US	-----

AB Catalysts suitable for the oxidation of aromatic hydrocarbons, and other compds., such as paraffins and **olefins**, each with at least 3 C atoms, aldehydes which yield **carboxylic acids**, and ketones. O-containing compds. are processed from molten catalytic vanadyl vanadates (single solid phase or a mixture of solid phases, each phase containing at least

1 of the metals Ti, Ag, K, Na, Li, or Rb, together with the oxides of V where part of the V atoms have a valency of 2 to 4) by impregnation onto a porous, heat-resistant, solid **support**, such as Al2O3, SiO2, SiC, or firebrick. The preferred catalysts are **supported** Ti vanadyl vanadate and K; in these, the M2O:V2O5 molar ratio, where M is K or Ti, may be 0.001:1-0.3:1, but is conveniently 0.05:1. Thus, 1.29 g. Ti2CO3 and 10 g. of V2O5 were fused together and impregnated on particles of α -Al2O3 by dipping. A mixture of 21.2 g. o-xylene **vapor** and 480 l. air was passed over 50 ml. of the Ti vanadyl vanadate **supported** on α -Al2O3. At the operating temperature of 500°

the o-xylene was converted to phthalic anhydride with a conversion of 64.1% and a pass yield of 35.5%.

L5 ANSWER 49 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1960:118497 CAPLUS
DOCUMENT NUMBER: 54:118497
ORIGINAL REFERENCE NO.: 54:22706a-f
TITLE: Addition of dinitrogen tetroxide to camphene and trans-stilbene; the effect of oxygen

AUTHOR(S): Stevens, Travis E.
CORPORATE SOURCE: Rohm and Haas Co., Huntsville, AL
SOURCE: Journal of the American Chemical Society (1959), 81, 3593-7
CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 54:118497

AB When an excess of N2O4 was added in a dry N stream to an Et2O solution of camphene, the products isolated were: 5% dl- α -nitrocamphene (I), m. 64-5°; 35% 3-(nitromethyl)-3-hydroxycamphenilane (II), m. 37-9°; 30% 3-(nitromethyl)-3-nitrocamphenilane (III), m. 141-3°; and a trace of a nitrate **ester**, probably 2-nitro-10-nitrocamphene (IV). II (formed by the hydrolysis of 3-(nitromethyl)-3-nitrocamphenilane) and III were the usual products of homolytic reaction of N2O4 with **olefins**. Treatment of II and III with a solution of KOH in 50% EtOH 30 min. gave I, 59 and 56%, resp. Similarly, base treatment of the crude camphene-N2O4 mixture gave I in 40% yield. With O as the **carrier gas**, the camphene-N2O4 reaction gave a mixture of products including 3-6% IV, 20-8% 3-isopropylidenecyclopentyl nitromethyl ketone (V), m. 76-7°, 30% nitroacids of undetd. structure, and a total of 6% II or III or both. The structure of V was established by its base-catalyzed cleavage to 3-isopropylidenecyclopentanecarboxylic acid (VI), m. 50°. VI was found to be identical with camphoic acid (prepared by the hydrolysis of the nitrile produced in the camphenilone oxime-AcCl reaction); ozonolysis of VI produced cyclopentanone-3-**carboxylic acid**, confirming the identity. The UV spectra of V had: in EtOH, 230 m μ (ϵ 3200) and 316 m μ (ϵ 660); in 50% EtOH-50% 0.1N NaOH, 234 m μ (ϵ 4500) and 331 m μ (ϵ 19,600). When an excess of N2O4 was swept with N into an Et2O solution of stilbene, the products (after hydrolysis of the nitronitriles) included 20.8% meso- α, α' -dinitrobenzyl, m. 235-7°, 32.2% dl- α, α' -dinitrobenzyl, m. 149-51°, 10.4% erythro- α -hydroxy- α' -nitrobenzyl (VII), m. 99-100°, and 12.6% threo- α -hydroxy- α' -nitrobenzyl (VIII), m. 105.5-6.3°. With O as

carrier gas, the products varied with minor changes in reaction conditions producing from 13.8 to 28.8% mixture consisting of 44% VII and 56% VIII, from 5.4 to 24.9% of a mixture consisting of 65% erythro- and 35% threo- α -nitro- α' -nitrobenzyl (threo- m. 96°, erythro- m. 165°), from 14.8 to 24.2% α -nitro- α' -phenylacetophenone (IX), m. 75.0-5.5°, 0-7.4% PhCO2H, and 0-1.1% PhCHO. Treatment of IX with alc. NaOH produced PhCO2H and PhCH2NO2. It was suggested that the nitroalkyl radicals reacted with O to form nitroperoxy or nitroperoxynitrate radicals.

L5 ANSWER 50 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1959:121688 CAPLUS
DOCUMENT NUMBER: 53:121688
ORIGINAL REFERENCE NO.: 53:21750g-i, 21751a-f
TITLE: Mechanism of the Michaelis-Arbuzov reaction: olefin formation
AUTHOR(S): Garner, Albert Y.; Chapin, Earl C.; Scanlon, Patricia M.

CORPORATE SOURCE: Monsanto Chem. Co., Springfield, MA
SOURCE: Journal of Organic Chemistry (1959), 24, 532-6

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE:

Journal

LANGUAGE:

Unavailable

OTHER SOURCE(S):

CASREACT 53:121688

AB

SUPPORT is given for the mechanism of the Michaelis-Arbuzov

reaction in terms of the formation of a quasi-phosphonium salt intermediate. The production of **olefin** and dialkyl phosphonate, (RO)₂POH, is shown to be a general phenomenon when an α -haloalkane which has an activating group on the β -C is treated with a trialkyl phosphite. The formation of these products is explained in terms of an intramolecular β -elimination involving the quasi-phosphonium salt intermediate. PhCH₂CH₂Br (92.5 g.), 74.9 g. NaI, and 50 ml. Me₂CO refluxed overnight gave 92 g. PhCH₂CH₂I (I), b.p. 117°, n_D²⁰ 1.5945. Triphenyl- β -phenethylphosphonium iodide (5 g.) left overnight with excess 10% aqueous NaOH gave 2.5 g. diphenyl phenethylphosphonate (II), needles, m. 75-6° (hexane). I (73 g.) and 146.2 g. P(OPh)₃ heated 5 days at 128°, the mixture mixed with Et₂O and Me₂CO until it was cream-colored, and the solvent removed gave 28.3 g. triphenylphenethylphosphonium iodide (III), m. 154-7° (sealed tube), gave an immediate precipitate with AgNO₃ which was insol. in dilute HNO₃.

MeCHBr₂ (7.52 g.) refluxed 1.5 hrs. at 131° with addition of 183 g. P(OPh)₃, and heated 3.5 hrs. at 145°, 110.8 g. EtBr removed, and the residue fractionated gave 509.6 g. unreacted dibromide. The first-boiling fraction of 33.7 g. consisted of a mixture of di-Et vinylphosphonate, di-Et phosphonate, and di-Et ethylphosphonate as shown by infrared analysis and vapor phase chromatography.

Subsequently 127 g. di-Et β -bromomethylphosphonate (IV), b.p. 90°, n_D²⁰ 1.4564, was obtained. An intermediate fraction (7.7 g.), n_D²⁰ 1.4473, which contained some tetraethyl ethylenephosphonate, was followed by the sudden evolution of 13.4 g. of material which dropped the head temperature to 30°/2 mm., and had n_D²⁰ 1.4350, shown to be pure di-Et vinylphosphonate (V). IV (50.7 g.) heated to 157°, and 35.3 g. distilled P(OPh)₃ (VI) added dropwise in 3 hrs., the heating continued to a total of 6 hrs., and 17.9 g. EtBr removed, and vacuum distilled gave 28.8 g. of low boiling material of approx. 64.3% diethyl phosphonate, 25.8% V, 6.9% diethyl ethylphosphonate (VII), and 2.9% of unknown material. A higher boiling fraction consisted of 16.2 g. tetraethyl ethylenephosphonate (VIII), b.p. 151-7°, n_D²⁰ 1.4397. Redistd. VI (90 g.) added dropwise during 3 hrs. to 100 g. Et β -bromopropionate at 135°, the mixture heated 5 hrs., and 48.4 g. EtBr removed during the heating. Vacuum distillation gave 21.9 g. material, b.p. 66-86°.

whose infrared spectrum showed it to be di-Et phosphonate and probably VII. 80 g. Et 3-diethylphosphonopropionate, b.p. 114-15°, n_D²⁰ 1.4301, and 24.1 g. poly(ethyl acrylate), n_D²⁰ 1.3975. The acrylate monomer and polymer were identified by their infrared spectra. PNCCH₂CH₂Br (100 g.) heated to 165°, treated dropwise with 90.9 g. VI, the temperature rose slightly, and further heated for approx. 20 hrs. gave 33.6 g. EtBr; the mixture distilled in vacuo gave 43.9 g. of 1st fraction containing diethyl phosphonate, VII, and unreacted material; the 2nd fraction was mostly VII, 3.9 g.; and the 3rd fraction of 68.8 g. consisted of di-Et phenethylphosphonate, b.p. 3 144-7°, n_D²⁰ 1.4925. The residue was dissolved in C₆H₆ and 3.2 g. polystyrene was precipitated by MeOH. The cold

trap

contained 10.4 g. impure styrene. VIII heated 5.5 hrs. at 211° and the material pumped down and heated gave no low-boiling materials. II (8.1 g.) was heated 2 hrs. at 240°/0.15 mm. without decomposition and heated again 4.5 hrs. at 250-305°/5 mm., still without decomposition II was recovered. Then the sample was heated at atmospheric pressure up to 390° to give a trace of H₂O, and a viscous brown residue, which was strongly acidic. The odor of styrene was strong in the trap but no styrene was isolated. The infrared spectrum showed polystyrene to be absent. III (10 g.) was dried overnight in vacuo, and heated 4 hrs. at 210-20°/0.15 mm.; the Dry Ice traps contained a small amount of liquid which had the odor of PhI and hinted at the presence of styrene.

Vapor phase chromatography showed the presence of a little styrene. The residue was crystallized to give II.

L5 ANSWER 51 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1955:56835 CAPLUS

DOCUMENT NUMBER: 49:56835

ORIGINAL REFERENCE NO.: 49:11003c-e

TITLE: Ketones

INVENTOR(S): Natta, Giulio; Ercoli, Raffaele

PATENT ASSIGNEE(S): "Montecatini" Societa Generale per l'Industria

Mineraria e Chimica

PATENT TYPE: Patent

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION: Unavailable

PATENT NO. IT 475040

KIND DATE APPLICATION NO. DATE

IT 475040 19521009 IT

AB The process involves a reaction of **olefins** (including mixts.

with paraffins) with CO (containing up to 10% H as an impurity) and primary or secondary alcs., at 150-250° and 150-500 atmospheric pressure, in the presence of Co-containing catalysts (fine Co. Raney Co. Co on an inert carrier, Co carbonyl or hydrocarbonyl, or other Co compds. which decompose to the ones mentioned). Examples are: (1) Secondary BuOH (I) 87 g., ethylene (II) 41.5 g., Raney Co (III) 4 g., and CO 140 l. in a 1-1. autoclave are shaken and warmed to 200-10° for 5 hrs., and cooled. Gas analysis shows that 25% of II did not react, while the liquid phase was shown to contain ketones 42.5, II 43.5, and propionaldehyde (IV) 3%. The yield of EtCO (V) was 65% based on the weight of II reacted: (2) MeOH 75 g., II 25 g., III 3 g., and CO 180 l. were treated in the autoclave at 190-95° for 85 hrs. to give: unreacted II 19%, and V 53%. Me propionate 20, and IV 7% (based on the weight of II reacted: (3) MeOH 220 g., propylene (VI) 84 g., III 16 g., and CO 103 l. at 200-10°, and 150-300 atmospheric yielded unreacted VI 49% and C7-ketones 27.5, Me butyrate and isobutyrate 33% based on the weight of VI reacted.

L5 ANSWER 52 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1939:32391 CAPLUS

DOCUMENT NUMBER: 33:32391

ORIGINAL REFERENCE NO.: 33:4579b-i, 4580a-h

TITLE: Oxidation of ethylenic hydrocarbons with selenium dioxide

AUTHOR(S): Guillemonat, Andre

SOURCE: Annali di Chimica Applicata (1939), 11, 143-211

CODEN: ACAPAR; ISSN: 0363-1037

DOCUMENT TYPE: Journal

OTHER SOURCE(S): CASREACT 33:32391

AB I. Pulverized SeO₂ (from the oxidation of Se in the presence of nitrous

vapors) is slowly introduced into a mixture of AcOH, Ac₂O and excess hydrocarbon while stirring vigorously so that the reagent does not settle to the bottom of the flask. The SeO₂ disappears slowly and Se ppts. The reaction mass turns yellow, orange and finally deep brown. When all the SeO₂ has reacted, the mixture is refluxed for at least 10 h., cooled, the Se separated, and the excess AcOH and Ac₂O are removed by washing with H₂O. The crude colored product is steam-distilled and the residue pyrolyzed at atmospheric pressure or under a vacuum. The pyrolysis products differ from the products in the distillate. Oxidation in a basic medium (pure pyridine) was unsuccessful. Various solvents were tried, including EtOH, xylene, H₂O, but AcOH proved most favorable both as to yield and product quality. In all instances the double bond is preserved. II. Oxidation of completely substituted ethylenic C atoms. 1. Aliphatic hydrocarbons. In all instances an appreciable amount of the initial hydrocarbon is recovered unattacked.

The oxidation occurs on the C in the α -position to the substituted atom; there is formed an alc. of the structure $\text{RCH}(\text{OH})\text{CMe}_2\text{CHMe}$. In order of decreasing ease of oxidation the radicals are CH_2 , Me, CH. The rates of oxidation are so different that only 1 of the possible alcs. forms if the radicals in the α -position are different. 2-Methyl-2-butene forms the acetate of 2-methyl-2-buten-1-ol, which on saponification with $\text{Ba}(\text{OH})_2$ yields the alc., b760 136-8°. Likewise, 3-methyl-2-pentene forms 3-methyl-3-penten-2-ol, b18 34-6°. 2,3-Dimethyl-3-pentene (from the oxidation of 3-methyl-3-pentene by SeO_2 to 3-methyl-3-penten-2-ol, bromination by PBr_3 and reaction of the bromide with MeMgBr), b760 91°, d421 0.720, nD21 1.4135, oxidizes to 2-isopropyl-2-buten-1-ol, b760 65-7°. 2,2,3-Trimethyl-3-pentene forms 2-tert-butyl-2-buten-1-ol, b22 82°. 2-Methyl-2-pentene yields 2-methyl-2-penten-1-ol, b14 at 61-3°. 3-Phenyl-3-pentene forms 3-phenyl-3-penten-2-ol, b20 127-30°. 2. Cyclic hydrocarbons. It was observed that oxidation will occur in the α -position to the most substituted C atom and also in the cycle if it is possible. Oxidation of the α -phenyl group leads to conjugated dienes by way of the dehydration of tertiary alcs. Conjugated dienes also result from the oxidation of hydrocarbons possessing cyclic bi-tertiary double bonds. Thus, 1-ethylcyclohexene yields 30% of a liquid acetate of 1-ethylcyclohexen-6-ol which hydrolyzes to the corresponding alc., b12 82-83°. 1-Ethylcyclopentene forms 1-ethylcyclopenten-5-ol, b20 74-5°. 1,6-Dimethylcyclohexene oxidizes to 5 fractions, b. 127-8°, 130.5°, 132.5°, 134° and 135.5°. Each fraction contains o-xylene, a quantity of a liquid b760 132.5°, nD20 1.4682, d420 0.832, and also 2,3-dimethyl-1,3-cyclohexadiene, identified by its maleic anhydride derivative, m. 122-3°. 1,2-Dimethylcyclohexene oxidizes to form 2 fractions. The 1st consists of o-xylene, and 2,3-dimethyl-1,3-cyclohexadiene, identified as above and also by its derivative with $(\text{tptbrom})\text{CCO}_2\text{Me}$ 2, b760 165-70°, which is hydrogenated to 4,5-dimethyl-1,2-phthalic acid, m. 196°. The 2nd fraction is a mixture of 2 acetates, one ethylenic and one dienic, yielding, on saponification, alc. products b23 95°, nD19 1.500, d419 0.971, but not in sufficient amts. to identify. III. Oxidation of hydrocarbons neither of whose ethylenic C atoms is completely substituted. 1. Aliphatic hydrocarbons. Again, oxidation occurs in the α -position to the ethylenic C. The CH_2 radical oxidizes more readily than Me. A double bond at the end of the chain is as active as a bi-secondary bond but due to transposition a primary alc. forms instead of a secondary alc. If a CH_2 radical is present on each side of the ethylenic C, both are oxidized and a mixture of alcs. forms which it is impossible to sep. Thus, 2-pentene forms the acetate of 2-penten-4-ol which is saponified to the corresponding alc., b760 118-21°. 1-Hexene yields 2-hexen-1-ol, b760 156°, with a small amount of 1-hexen-3(?)ol. 4-Nonene on oxidation yields a liquid, b15 89-91°, which on saponification forms a viscous liquid, b11 85-7°, and which is hydrogenated to a substance b18 90-1°. All attempts to prepare crystalline derivs. have failed. Presumably, the product is a mixture of nonenols formed by oxidation of the two CH_2 groups in the α -position to the ethylenic C atoms. Likewise, 3-nonene forms an acetate, b17 99-101°, which is saponified to nonenols, b15 93-5°, and hydrogenated to a mixture of nonanols, b17 93°. As in the preceding case no crystalline derivs. could be prepared. 2. Cyclic hydrocarbons. While not so reactive as compds. with double-linked tertiary C atoms, cyclic hydrocarbons with bi-secondary ethylenic bonds do produce yields as high as 30-40%. Again the α -position is attacked. The CH_2 group is more active than the CH. Again, also, both possible CH_2 groups are attacked simultaneously. Transpositions of the allylpropenyl type occur readily. Thus, cyclohexene yields the acetate of 1-cyclohexen-3-ol, b15 68-70°, saponifying to the corresponding alc., b15 67°, identified by its phenylurethane, m. 106 5-7.5°. Oxidation by CrO_3 forms the corresponding ketone whose semicarbazone m. 161°. 3-Methylcyclohexene likewise produces 6-methylcyclohexen-3-ol, b20 88-90°, which is hydrogenated to 4-methylcyclohexanol, b760

169°. Small amts. of MePh, 4-methylcyclohexene and 4-methylcyclohexen-3-ol also result from the oxidation of 4-methylcyclohexen-3-ol, b22 81-3°, the 1st predominating. The 3 corresponding alcs., formed on saponification, b6 63°. Another fraction, b22 82-3°, is obtained which is also a mixture of the 3 acetates above but with the 5-Me derivative predominating. For all the oxidation products the author gives b. p., d., n, mol. refraction, as well as m. ps. of the various derivs. mentioned and in some cases C and H analyses. IV. Mechanism of the oxidation. The postulated mechanism may best be represented by means of the following equations in which R is a radical containing the ethylenic bond: (1) $4\text{RCH}_2\text{H} + \text{SeO}_2 \rightarrow (\text{RCH}_2)_4\text{Se} + 2\text{H}_2\text{O}$ (2) $(\text{RCH}_2)_4\text{Se} + \text{H}_2\text{O} \rightarrow (\text{RCH}_2)_2\text{Se} + \text{RCH}_3 + \text{RCH}_2\text{OH}$ (3) $(\text{RCH}_2)_2\text{Se} + \text{H}_2\text{O} \rightarrow \text{RCH}_2\text{OH} + \text{RCH}_3 + \text{Se}$ (4) $(\text{MeCH}_2)_2\text{Se} + \text{CH}_2\text{:CHCMe:CH}_2 + \text{Me}_2\text{C:CHMe} + \text{Se}$ This proposed mechanism explains (1) the fact that a portion of the original hydrocarbon is recovered although the theor. amount of SeO_2 is used, (2) the formation of an alkyl acetate even at low temps. since HOAc may supplant HOH in reaction 2 above, (3) the formation of an ether oxide in an absolute alc. medium by a mechanism similar to that which furnishes the acetate and (4) the formation of dienes with conjugated systems. This mechanism is supported by exptl. data on the oxidation of trimethylethylene in benzene. V. A complete table of the Raman spectra of all the compds. mentioned in the previous chapters is given, 38 compds. being listed in all.

=> LOG HOLD	SINCE FILE	TOTAL
COST IN U.S. DOLLARS	ENTRY	SESSION
FULL ESTIMATED COST	243.27	244.11
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
CA SUBSCRIBER PRICE	ENTRY	SESSION
	-42.75	-42.75

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